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The Ohio State University

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Plastic Instability and Hydrogen Embrittlement in Steels

DISSERTATION

Presented in Partial Fulfillment of the Requirements for
the degree Doctor of Philosophy in the Graduate
School of The Ohio State University

By
Vaidyanath Bharata Rajan, B. Tech., M. S

* * * * *

The Ohio State University
1984

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This thesis is dedicated to my mother without whose encouragement and support I would not have been able to achieve this level of education.
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INTRODUCTION

Hydrogen embrittlement phenomena have been observed and studied in a number of different systems for the last hundred years, with the result that a number of seemingly diverse observations have been reported. To explain these results a number of proposed mechanisms and models, some of them even conflicting with each other, have come into vogue. As a result, no single unified theory exists today to explain hydrogen effects in metals.

Of late, two main points of view seem to be emerging out of the medley of mechanisms in the literature. One of them is hydrogen induced strain controlled fracture and the other is hydrogen induced stress controlled fracture.

In strain controlled fracture, the most significant and important effects are those of hydrogen promoting plastic instability and ductile fracture. To understand this effect, a significant understanding of plastic instability has to be achieved. The current plasticity theories predict that when a smooth, isotropically hardening yield surface is assumed, the critical stresses required to cause strain localization and instability are of the order of the
shear modulus. But when the concept of a corner or vertex on the yield surface is invoked, more realistic values of stresses and strains for instability are attained. This gives rise to the idea of a material undergoing flow localization when it encounters a corner on its yield surface. Hydrogen has been observed to enhance localized plasticity and fracture in iron and steel. So it becomes vital to gain a significant understanding of the process of plastic instability, to understand hydrogen induced "strain controlled" fracture.

On the other hand, hydrogen has been shown to cause seemingly very brittle fractures in medium and high strength materials, and these have been termed "stress controlled" fracture. Here the stress triaxiality, concentration of segregated impurities, and the strength level of the steel have been cited as important factors influencing this mode of fracture. But even in these seemingly brittle fractures, localized plasticity has been observed. So the effect of hydrogen in enhancing localized plasticity even in such cases cannot be ruled out.

This research effort was undertaken to gain a better understanding of these two effects. The materials selected were AISI 1045 and 1090 steels which are simple yet microstructurally versatile materials, so that a number of different combinations of microstructures and strength levels
could be obtained. U-notched specimens of 1090 steel were tested in the high toughness spheroidized condition in four point bending. U-notched specimens were used to reduce the effect of stress triaxiality and to shift the point of maximum triaxial stress, away from the notch surface, into the bulk. This enabled study of instability and fracture without the overwhelming effect of triaxial stresses at the notch root. The four point bending mode was selected so that notch deformation could also be studied in compression where the complicating effects of void formation are excluded. The above setup was thus helpful in studying strain controlled fracture.

Quenched and tempered 1045 and 1090 steels were also used in two different heat treated conditions. In one set of experiments, both types of steels were tempered in the tempered martensitic embrittlement range. In another set of experiments, these steels were tempered outside the tempered martensitic embrittlement range. These two conditions were used to study the effect of segregation, yield strength and carbon content on instability and strain controlled fracture, stress controlled fracture and the partitioning of hydrogen effects between these fracture modes.
Chapter I
LITERATURE SURVEY

1.1 PLASTIC INSTABILITY
Most metals have been recognized to undergo some amount of plastic deformation before failure. In ductile metals subject to an increasing plastic deformation field, it is quite common to have that homogenous field give way to bands of intense localized deformation. When this happens, especially underneath a microstructural or geometric discontinuity (like a notch), the conditions to initiate and propagate a fracture are more readily attained.

The most well known form of this strain localization or plastic instability as it is known is the "necking phenomenon" in a tensile test. In this case, the geometric softening caused by an increase in stress resulting from a decrease in the cross sectional area of the specimen during necking, exceeds the increase in the load carrying capability of the material caused by strain hardening. This leads to very localized deformation in the neck and eventual failure.
Plastic instability, in general, has been found to occur in a number of different systems. The formation of Luder bands during plastic deformation of steels is a good example of transient instability. High strain rate processes like shearing [1], punching [2], high impact erosions [3], and high strain rate extrusion [4] also exhibit a form of plastic instability. In these cases the heat generated during plastic deformation has insufficient time to be dissipated because of the high strain rate deformation. The eventual adiabatic temperature rise in the deformed region promotes localized softening, giving rise to what are called adiabatic shear bands. This local temperature rise can also induce metallurgical instabilities like reversion of martensite [13], redissolution of precipitates [5] etc. More information on this topic can be found in comprehensive reviews by Bedford [4] and Rogers [6].

The models of instability which are relevant to this work are the ones discussed by Spretnak and coworkers [7]-[12], Rice and coworkers [14]-[17], Asaro [15], [18], Hutchinson et al. [19], [20], and Needleman [21], in which material instability to plastic flow is considered. The models of McClintock et al. [22], [23], and Yamamoto [24], who consider geometric instability during plastic flow, as evidenced by void formation and growth, are also reviewed.
Examples of the above modes of instability can be found in the works of Beavers and Honeycombe [25], who observed localization of deformation in coarse slip bands formed within a diffusely necked region of their specimen during ductile fracture of single crystals. Price and Kelly [26] also observed strain localization in Al single crystals. Hahn and Rosenfield [27] have observed the formation of long localized bands in peak aged Al alloys leading to lower toughness in the material. French and Weinrich [28] found bands of intense shear deformation in the ductile fracture of low strength α-brass. The above works are good examples of material instability in high and low strength materials. But there are examples where the relative contributions of material and geometric softening in fracture are less clear. Cox and Low [29] observed shear bands, containing microvoids, connecting cavities formed at brittle inclusions. Rogers [30], and Bluhm & Morissey [31] have observed zones of intense shear, containing voids which are coalescing. In these works, it is difficult to determine whether the formation of voids leads to plastic instability via geometric softening, or whether the plastic flow localization promotes void formation and growth. To obtain a better understanding of the process of plastic instability, it is worthwhile to review some of the theoretical models in the literature.
Spretnak [9] has followed the idea of a Dutch plastician, van Iterson [32], and considered the possibility of the material attaining a plasticized state (between a solid and a liquid), where all the strain gets concentrated in localized bands in which the material becomes ideally plastic. In particular, these plasticized zones are the characteristic surfaces of pure shear with no extensional strain. It is significant that in the cases of plane stress and plane strain, these surfaces coincide with the characteristic surfaces of slip line field theory, where the shear strain is a maximum.

Spretnak [9] has invoked the concept of the tangential velocity discontinuity of plasticity theory [33] to interpret plastic instability in pure shear. The characteristic or discontinuity surface, which is a slip line in two dimensions, cannot allow a normal velocity or displacement discontinuity across it due to continuity restrictions. But it can allow a tangential velocity or displacement discontinuity, and when this is activated, one of two things can happen. If the discontinuity is damped out, the deformation is stable and fracture does not occur. But if the discontinuity persists, plastic flow gets concentrated in directions of pure shear, the material becoming rigid on either side of the characteristic surface, leading to instability. Spretnak pointed out that the presence of
(a) a free surface (b) a stress gradient (c) low strain hardening, promoted the occurrence of such instabilities.

Argon [34] and Hornbogen et al. [35] have invoked the concept of strain softening or negative strain hardening as a necessary condition for inhomogeneous deformation to take place, which seems to be a very restrictive condition for plastic instability to occur.

But detailed analytical and experimental work by Asaro and coworkers [15],[18] shows that in ductile single crystals, shear localization can occur with low positive strain hardening without the need for an ideal plastic or strain softening state. They show that when the plastic hardening modulus $h$ for the slip system has fallen to a critical value $h_{cr}$, shear localization can occur. Their analysis, based on a smooth yield surface during single slip, has included the effect of nonnormality and nonSchmid effects. Microscopic processes like cross slip have been cited as specific examples where stresses other than Schmid stresses influence the constitutive law governing incremental shear, and $h_{cr}$ which is sensitive to the constitutive description of the material has been shown to be positive at the onset of shear localization. Chang and Asaro [18] have provided experimental evidence of geometric softening effects brought about by lattice rotations in a shear band. Here the shear band plane rotates towards the primary slip
plane increasing the critical resolved shear stress along itself, thus producing nonnormality during plastic flow. But all these investigations have been restricted to single crystals, and effects of inherent material or geometric inhomogeneity in the material have not been considered.

Storen and Rice [17] analyzed the biaxial stretching of thin sheets and observed that when the $J_2$ flow theory of plasticity with a smooth yield locus is used, strain localization cannot occur in biaxial tension. However, they showed that a constitutive model of a pointed vertex on the yield locus, using the $J_2$ deformation theory of plasticity for proportional loading, gives values of strain for the onset of localized necking in good agreement with experimental observations. In fact, Hill [36] and Hutchinson [37] have shown that the vertex formation on a yield locus is a common feature in polycrystalline materials, when slip in each grain is governed by Schmid's law.

Rudnicki and Rice [16] considered the localization of deformation into a shear band as a consequence of an instability in the constitutive description of the material. This instability is marked by the loss of ellipticity of the field equations leading to a bifurcation point in the constitutive response. This leads to nonuniform deformation in a planar band surrounded by regions undergoing homogenous deformation. They considered this instability as
brought about by nonnormality of plastic strain increments due to pressure dependent yielding and yield vertex effects. The two effects are shown schematically in Figures 1 and 2. Consideration of these two effects resulted in more realistic predictions of localization strains. But they did not consider the effect of initial inhomogeneities in the material.

Figure 1: Effect of pressure on the yield surface
Figure 2: Formation of a vertex on the yield surface

Tvergaard [38] suggested that instead of isotropic hardening, kinematic hardening behavior of the material during plastic strain increments, especially for a high strain hardening material, should be considered. In kinematic hardening, the yield surface does not expand but instead translates in stress space. Using this assumption
and assuming the presence of initial inhomogeneities, he predicts a bifurcation strain in a biaxially stretched sheet, which agrees reasonably well with the forming limit curves. But yield vertex effects are not specifically included in his analysis.

Needleman [21] investigated bifurcation of plastic flow in a rectangular block subjected to plain strain tension and compression. He considered an incompressible material, which is characterized by an incrementally linear constitutive law and does not obey normality to study elliptic, hyperbolic and parabolic regimes of the governing field equations. He noted that in plane-strain tension, ellipticity is lost prior to the attainment of maximum load and shear bifurcation and short wavelength diffuse modes become available simultaneously when,

$$\sigma/2G < \delta^2/(2 - \delta^2)$$  (1)

Here $\sigma$ is the applied stress, $G$ the instantaneous shear modulus and $\delta$ is a coupling parameter between shear strain and hydrostatic pressure. But if,

$$\delta^2/(2 - \delta^2) < \sigma/2G < \delta$$  (2)

then the maximum load is attained prior to loss of ellipticity, and diffuse necking precedes shear band bifurcation. If normality is obeyed, then an infinite number of bifurcation modes in the elliptic regime are available.
before loss of ellipticity, as was also found by Hill [40] and Hutchinson [37]. In plane strain compression, depending on the value of the instantaneous tangent modulus and specimen dimensions, diffuse bifurcation may or may not precede shear band bifurcation. But when the effect of the yield vertex is included, the instantaneous shear modulus is decreased such that diffuse bifurcation precedes the onset of localized shear. Further details on this topic can be obtained in Needleman’s analysis.

The bifurcation analysis that is most easily applicable to the work in this thesis is that of Hutchinson and Tvergaard [19], [20]. They studied the development of surface instabilities on traction-free surfaces of rate independent solids. Of particular relevance is the bifurcation analysis using the $J^2$ deformation theory for hyperelastic (path independent) solids to predict the onset of surface instabilities. This theory invokes the concept of a vertex developing on the yield surface giving rise to a bifurcation point in the constitutive response. This signifies the beginning of localized plastic flow. To describe the post-bifurcation response however, the $J^2$ deformation theory is not adequate, and so the $J^2$ corner theory of plasticity proposed by Christofferson et al [41] has been used to describe the evolution of the growth of the surface instabilities. The $J^2$ corner theory is a modification
of the $J_2$ deformation theory such that, the instantaneous moduli in the corner theory coincide with those of the deformation theory for nearly proportional loading increments, but increase smoothly to the elastic moduli values for nonproportional loading which occurs during the post bifurcation deformation.

The condition for surface bifurcation is given by,

$$\sigma_2/4\mu^* = 1 + \sigma_2/4\mu^*[(2\mu - \sigma_2)/(2\mu + \sigma_2)]^{1/2}$$  (3)

where $\mu$ and $\mu^*$ are the instantaneous moduli for shearing parallel and at $45^0$ to the coordinate axes and $\sigma_2$ is the stress in the loading direction.

$$\mu = (1/3)E_s(\varepsilon_1 - \varepsilon_2)\coth(\varepsilon_1 - \varepsilon_2)$$  (4)

$$\mu^* = (1/3)[E_s - (E_s - E_t)(\cos\alpha + \sin\alpha)^2(1 + \sin\alpha/2)^{-1}]$$  (5)

where $E_s$ and $E_t$ are the secant and tangent modulus of the uniaxial true stress-true strain curve, $X$ and $\alpha$ define the magnitudes of the true strains $\varepsilon_2$ and $\varepsilon_3$

$$\varepsilon_2 = X \cos\alpha, \quad \varepsilon_3 = X \sin\alpha$$  (6)

They point out that the onset of these surface instabilities will occur before the onset of shear bands as shown in Figure 3. The results of their study on the nonlinear growth of these instabilities is presented in Figure 4. They further conclude that the presence of large initial surface imperfections causes a faster growth of these
instabilities than would be observed for an initially smooth surface. However their treatment does not include plastic dilatancy or nonnormality effects on the constitutive response of the material.

Hutchinson et al [20] also studied the initiation of shear bands in incompressible solids in plane strain. The bifurcation condition for the onset of a shear band with orientation $\psi$ with respect to the surface is,

$$\left( \mu - \frac{\sigma_2}{2} \right) \tan^4 \psi + 2(2\mu^* - \mu) \tan^2 \psi + \left( \mu + \frac{\sigma_2}{2} \right) = 0$$  

(7)

where the symbols have the same meaning as mentioned before. For loss of ellipticity to be possible, the above equation must have real solutions in $\tan \psi$, which is possible when,

$$\mu > \frac{\sigma}{2}, \quad 2\mu^* \leq \mu - (\mu^2 - \sigma^2/4)^{1/2}$$  

(8)

A significant amount of work has also been done in studying the geometric softening contribution to plastic instability. In these studies the effects of void initiation and growth on plastic instability is modelled and some of these will be reviewed briefly.

Tvergaard [39] studied the influence of microvoid initiation and growth on the occurrence of instabilities near a surface, for a solid under plane strain conditions. He concluded that when strain controlled microvoid nucleation and growth was prevalent, the surface undulations do not pre-
Critical strains for surface bifurcations for $N = 0.5$. Outer curve marks onset of shear bands.

Figure 3: The onset of surface and shear bifurcation at different strains. From [19].
Fig. 7. Growth of initial waviness as function of overall tensile strain $\epsilon_t$ for $J$-corner theory with $\epsilon_n = 0.005$ and $N = 0.1$.

Fig. 8. Growth of initial waviness as function of overall compressive strain $\epsilon_c$ for $J$-corner theory with $\epsilon_n = 0.005$ and $N = 0.1$.

Figure 4: The growth of surface instabilities with strain. Here $b$ is the half amplitude of the surface wave and $l_0$ is the initial wavelength of the surface wave. From [19].
cede the onset of shear bands, contrary to the findings of Hutchinson et al [19]. He also concluded that if microvoid effects are more dominant than yield surface vertex effects, then bifurcation is predicted to occur earlier in tensile rather than in compressive loading and vice versa. He also numerically analyzed the effect of initial material imperfections, and found that shear band growth occurred earlier when plastic strain controlled nucleation was predominant, than when there are no imperfections. But when void nucleation was stress controlled, the onset of shear bands was delayed until after the loss of ellipticity of the field equations.

McClintock et al [22],[23] studied void growth leading to geometric softening, causing mechanical shear instability in a band. But they did not include bifurcation analysis of the constitutive plasticity laws during the instability. So they underestimated the critical strain for instability and fracture. Yamamoto [24] studied shear localization in materials containing voids. He considered geometric softening by void growth in materials containing initial imperfections. He included the constitutive relations and applied them to these materials to predict the onset of flow localization by softening due to void growth. He concluded that when the void concentration in an imperfection band is higher than in the surrounding regions, localiza-
tion sets in at lower strains. Localization also becomes more imperfection sensitive with an increase in the initial void volume fraction outside an imperfection. Shear localization is also enhanced by a decrease in the strain hardening coefficient of the material.

1.2 A BRIEF REVIEW OF HYDROGEN DEGRADATION MODELS

A number of different hydrogen degradation [42] phenomena have been identified over the years. To explain these observations a number of degradation models have been presented. These models have been introduced to account for certain observations and support certain points of view. Hence none of the models by themselves have emerged as a true general model for hydrogen embrittlement. Moreover, as Louthan has pointed out[50], a lot of the experiments are designed to point out weaknesses in other theories without providing support for a single model. Nevertheless, it is prudent to consider some of these theories, because as Hirth has pointed out [51], even though none of the models by themselves qualify as a general model, most of them under appropriate conditions could be part of a complex overall mechanism.
1.2.1 Internal Pressure Theory

Zapffe and Sims [52] and Tetelman et al [53] suggested that lattice hydrogen could collect in voids as hydrogen gas, building up high internal pressures in the voids. This internal pressure would aid the applied tensile stresses in promoting void or crack growth in the material. It was further suggested that dislocations could transport hydrogen [55],[56], to these voids thus building up internal pressures, even when the external hydrogen pressure is low. Thus Hancock and Johnson's results [54] which show evidence of crack propagation even at low external hydrogen pressures seem to support the above arguments. But it has been shown by Johnson and Hirth [57] that at room temperatures, a supersaturation factor of only about 2 or 3 can be obtained, though higher supersaturations can be obtained at lower temperatures. Besides at room temperature, only if the external hydrogen fugacity is high can substantial internal pressures be built up. Thus this model does not have applicability as a general mechanism.

1.2.2 Slip Softening Models

Beachem [58] noted that the torsional stress was lowered in 1020 steel in the presence of hydrogen. He proposed that hydrogen augments dislocation motion and thus enhances local plasticity at a crack tip. Lynch [59] noted the similarities in the features of the fractured surfaces in lig-
uid metal embrittlement and hydrogen induced intergranular cracking. He suggested that the effect of hydrogen is due to adsorption at defect sites such as crack tips. Hydrogen could then cause crack growth by enhanced injection of dislocations at the crack tip.

It has been observed [51] that hydrogen enhances screw dislocation mobility, dislocation injection at surfaces and promotes shear instabilities. So the enhancement of dislocation motion by hydrogen is quite established. But these softening models cannot account for the hardening effects [60] that have been observed, thus accounting for their loss of generality. Since enhanced dislocation motion would be required to start a crack near an inhomogeneity, it can be inferred that slip softening could be part of an overall degradation mechanism.

1.2.3 Hydride Formation Model

Westlake [61] suggested that hydride formation near a crack tip could induce stresses at the crack tip owing to the high molar volume of the hydride. This could promote the growth of the crack. Moreover, a stressed crack tip which has a hydrostatic stress field could stabilize a hydride near itself [62], even when the hydride would otherwise be unstable in the absence of stress. This model could be applied for hydrogen embrittlement of strong hydride formers like Nb. But for iron, hydrides are not stable even at
very high, GPA level, hydrogen pressures. So this model is not very applicable for irons and steels. On an atomic scale though, such an idea can be applied in a limited way, because high supersaturations of hydrogen have been observed near stressed crack tips [63].

1.2.4 Surface Energy Models

In the Griffith crack model, a part of the work done in fracture goes towards supplying surface energy for the surfaces created by the fracture process. Petch and Stables [64],[65], suggested that hydrogen adsorption lowers the surface energy of these new surfaces and thus reduces the work of fracture and promotes cracking. But surface energy is only a small part of the overall work of fracture, irreversible effects like plastic deformation accounting for a major part of the work of fracture. So this model grossly underestimates the work of fracture. This model cannot explain the occurrence of discontinuous cracking nor can it explain the reversibility of delayed failure on removal of stress. It also cannot account for the fact that oxygen, which has a higher heat of absorption, and thus a larger effect on lowering the surface energy, not only does not promote cracking, but also inhibits the hydrogen effect. This model could, however, apply on a microscopic scale as part of an overall complex process near a crack tip, when local crack propagation is nearly reversible [51].
1.2.5 Decohesion Theory

This theory, originally proposed by Troiano [66], considers the enrichment of hydrogen in the triaxial region of the notch, which lowers the cohesive strength of the atomic bonds leading to void formation and subsequent cracking. Oriani [67] used this theory to model hydrogen accumulation at crack tips due to the crack tip stress intensity. This idea has also been used to explain metalloid effects [68],[69], in hydrogen embrittlement. But this model cannot account for the ductile fracture mode observed in steels [58],[59],[70]. It also cannot explain hydrogen effects in promoting shear instabilities in the maximum strain region as opposed to the maximum stress region underneath a u-notch in spheroidized 1090 steel [70], [85]. But it can explain brittle fractures observed in high strength steels and the effects of metalloid impurities therein [68],[69]. The fundamental idea of decohesion inherent in this theory can, of course, be extended to explain enhanced void nucleation at inclusions and second phase particles and subsequent crack nucleation and propagation, because they all involve local atom bond breaking. In this way, this idea could be considered as part of an overall degradation model.
1.3 EFFECT OF HYDROGEN ON MECHANICAL PROPERTIES OF IRON AND STEEL

Hydrogen degradation phenomena have been observed to be varied and complicated. Efforts to study these phenomena have resulted in a lot of confusing and conflicting observations. The state of affairs could be best described in the words of Latanision et al [80] who said "Considering the volume of literature which has appeared on these subjects of HE and SCC, it is no surprise that evidence can be found to contradict virtually every point of view". Therefore, it is best to consider some of the general investigations and then concentrate on those which are of most relevance to this work.

Louthan and McNitt [50] listed a group of ten observations and they concluded that any valid hydrogen embrittlement mechanism must be compatible with them. Some of these are: hydrogen induces delayed failure in smooth, notched or precracked specimens with very little or no macroscopic strain preceding fracture; hydrogen exhibits temperature and strain rate dependence and is greatest at low strain rates and at a temperature where tensile ductility of uncharged specimens is highest; hydrogen induces ductility losses and slow crack growth with or without changes in fracture mode; deformation enhances hydrogen diffusion and causes enhanced outgassing of charged specimens and
enhanced adsorption by uncharged specimens; increases in gas pressure can increase crack growth. Yield strength increases in some alloys and decreases in others due to absorption of hydrogen; and gas phase hydrogen embrittlement can be inhibited by impurities such as oxygen and water vapor.

To understand the complex and confusing effect of hydrogen on steels it is necessary to comprehend the basic effect of hydrogen in a simpler system such as iron.

Matsui, Kimura and coworkers [71]-[73] have studied hydrogen effects on the deformation of iron of various purities, below room temperature. They observed that hydrogen induces reversible softening of very pure iron at 200\(^0\)K and above, when the charging current density exceeds a critical value. They attributed this to hydrogen enhanced mobility of screw dislocations The critical charging current was found to decrease with a lower strain rate, higher purity and lower temperature. The amount of hydrogen induced softening was found to be larger for the purer specimens. But above 273\(^0\)K, the hydrogen induced softening was found to be very small and was observed only for very pure specimens (RRR > 4000). No damage was observed on hydrogen charging of very pure specimens, damage (blistering) occurring only in the impure specimens (RRR =1800). Below 190\(^0\)K, hydrogen caused hardening, if the charging
current was greater than a critical value which was found to decrease with temperature. This hardening is attributed to increased drag on edge dislocations and edge kinks on screw dislocations. Above \(200^0\text{K}\), hydrogen induced hardening was observed when the impurity content was increased. They concluded that at \(200^0\text{K}\) and above, the intrinsic behavior of hydrogen is to promote softening by enhancing the mobility of primary screw dislocations. But at the same temperature range hardening would be observed if the impurity concentration were increased. This hardening behavior is attributed to hydrogen-impurity-dislocation interaction. Softening due to damage only occurs at high impurity and high hydrogen concentrations. These observations are best summarized in their schematic diagram as shown in Figure 5. As the temperature is raised it shrinks the intrinsic softening region, and hence at room temperature, hydrogen induced softening is rarely observed. Also at high deformations, screw dislocations no longer control the deformation as cell structures are formed, and hence hydrogen induced softening ceases to occur. Hydrogen also changed the fracture mode of the iron single crystals from a chisel point fracture to a mixture of cleavage and microvoid coalescence, with a concomitant reduction of 50% in ductility.
Figure 5: Effect of Hydrogen on Iron. From [71].

Shin, Meshii et al [75] have observed both softening and hardening behavior of iron when charged with hydrogen. They also attributed the softening behavior to enhanced dislocation mobility and the hardening behavior to impurity interactions. They observed that polycrystalline iron when charged with hydrogen at 200$^\circ$K showed intergranular fracture as opposed to transgranular quasicleavage in single crystals. The intergranular fracture was attributed to increased hydrogen interaction with the grain boundaries. Such intergranular fractures were also reported by Matsui et al [72] and Cornet et al [76] in very pure iron polycrystals.
Nagakawa et al [113], have studied the effect of hydrogen charging on creep of pure iron single crystals and polycrystals. They found enhancement in the creep rate in both single crystals and polycrystals. The fracture mode in single crystals remained unchanged, but that of polycrystals was changed to intergranular fracture in the presence of hydrogen. During the deformation of the hydrogenated samples, voids and cracks were observed but only in the later part of the deformation. Voids in the single crystals were formed as arrays parallel to the primary slip plane and in polycrystals were formed at grain boundaries. So these observations suggest that hydrogen enhances softening in pure iron and the voids that form later in the deformation can evidently enhance this softening.

The strongest evidence for hydrogen enhanced dislocation mobility has been provided by Birnbaum and coworkers [126], [127], who conducted in situ HVEM studies of hydrogen effects on deformation and crack growth in pure iron. They observed an enhancement of screw dislocation velocity and a concomitant increase in dislocation source activity at low hydrogen pressures (10^4 Pa). An in-situ study of crack propagation revealed enhanced dislocation emission from the crack tip on introduction of hydrogen gas. This lead to localized enhanced plasticity and thinning out of the material in front of the crack and eventual crack propaga-
tion. The fracture mode was generally transgranular and followed the (110) and (112) slip planes.

But contrary to these observations, hydrogen induced hardening in pure iron has also been reported. Asano et al [77] observed a reversible increase in flow stress of pure iron, ferritic steels and mild steel charged with hydrogen. Whatever softening they observed they attributed to damage due to hydrogen induced voids. But they used a very low charging current of $10 \text{A/m}^2$, and as was mentioned before, the hydrogen impurity interaction might have led to a hardening behavior. This can mask the softening effect.

There are a few other observations of hardening of iron by hydrogen [133],[78]. All the proponents of the hardening theory believe in an intrinsic hardening mechanism and a damage induced softening mechanism. Thus the controversy over whether hydrogen hardens or softens iron is quite evident in the literature. But the overall evidence is strongly in favor of an intrinsic softening effect and impurity or edge dislocation interaction induced hardening effect.

The controversy mentioned in iron is evident in steels, where the situation is more complex, because of the added variables of composition, strength and microstructure. A brief review of the reported effects of hydrogen on the yield strength of steels reveals the extent of this contro-
versy. Beachem [81] observed that the flow stress of 1020 steel in torsion was lowered by application of a cathodic current. Rogers [30] observed a lowering of the yield strength of 1020 steel by electrolytically charged hydrogen. Cracknell and Petch [83] also found a lowering of the yield strength for steels with varying carbon contents up to 0.6%, with hydrogen. Lee et al [84] obtained a lower yield strength in spheroidized 1090 steels electrolytically charged with hydrogen. Onyewuenyi et al [85] observed a lowering of the microhardness of spheroidized 1090 steel at medium and high hydrogen input fugacities.

Contrary to these results, Tobe et al [86] found an increase in yield strength and an enhancement of the yield point phenomena after gaseous charging with hydrogen. Oriani and Josephic [60] found no yield point effect on 1045 steels but observed an increase in flow stress with an increase in hydrogen input. Oriani [79] also pointed out that hydrogen broadens the core of the screw dislocation and thus increases the difficulty for cross slip. Since cross slip is necessary to bypass barriers such as carbides in steel, this reflects a general hardening behavior of hydrogen in steel. But core broadening also leads to a lower Peierl's stress and combined with the difficulty of cross slip, this could promote localized deformation and enhance instability. Seabrook et al [87] also observed an
increase of flow stress in 1020 steels with introduction of hydrogen.

These results indicate that both softening and hardening behaviors are possible in steel. The hardening was attributed to difficulty of cross slip. The softening, which no doubt will occur when voids and fissures are formed, can also occur due to enhanced dislocation emission, as has been demonstrated for pure iron.

Hirth [51] proposed a rationalization for this hardening-softening behavior. If charging has not resulted in damage, then softening during single slip is possible by the enhancement of double kink nucleation on screw dislocations and by dislocation injection at surfaces and interfaces. But if deformation is by multiple slip, then dislocation arrays are complex and work hardening occurs. This hardening could be due to difficulty of cross slip in hydrogenated iron, as evidenced by increased tangle formation at low strains [88]. Other reasons could be, increased difficulty for dislocation intersection involving core-core interactions between the hydrogen saturated cores, vacancy stabilization by hydrogen slowing down recovery processes, hydrogen augmenting carbon effects in core pinning and viscous core drag.

In general, hydrogen effects in steels are a complex function of the strength level [94], microstructure and
composition [92], hydrogen source [93], hydrogen transport [95],[55] and trapping [96], strain rate [97] etc. Thus there are a number of variables which have to be considered. This has resulted in an enormous amount of work in the literature. For clarity, it is best to consider those which are of most relevance to the work here.

It is quite common to think of hydrogen induced fractures as brittle and that the higher the strength of the steel, the more the tendency for hydrogen induced brittle fracture [94], [66],[98]-[102]. But hydrogen can induce ductility loss even by a ductile fracture mode, in low [90]-[105] and high strength steels [90],[59],[58] It is quite possible that localized plasticity and hydrogen play an important role in promoting ductility loss without changing the ductile fracture mode. Beachem [58] observed varied degrees of localized plasticity in cleavage, quasi-cleavage and ductile modes of hydrogen induced fracture in steels. More recently Thompson et al [90],[47] have also discussed hydrogen effects in promoting microscopic plastic flow. Observations which are most relevant to these ideas are those involving study of hydrogen induced/enhanced void initiation and growth in steels.

Oriani and Josephic [60],[106], have studied hydrogen effects on plastic flow, fracture and load relaxation in spheroidized and pearlitic 1045 steels. They suggested
that the hydrogen induced softening effects, as observed by an increase in load relaxation and decreased strain to fracture, were due to hydrogen induced microvoid initiation especially at carbide interfaces. They found no effect of hydrogen on void growth and that void growth did not occur preferentially along subgrain or grain boundaries.

Garber et al [105] studied hydrogen effects on void initiation and growth in unnotched round tensile bars of spheroidized 1018 and 1080 steels using the Argon [125] analysis. They observed no effect of hydrogen on void initiation and growth over most of the strain, before the final fracture process in both steels, in spite of a ductility loss of 20-25%. They studied void initiation and growth by measuring the areal density of voids and the average size of a void as a function of strain between necking and fracture. They found that hydrogen affected void initiation and growth only during the link up stage during the final fracture process. This observation is interesting in connection with the interactive role of plastic instability and hydrogen, because shear localization has been observed in tensile specimens after diffuse necking as observed by Anand et al [110]. Shear localization has also been observed in u-notched bend specimens of 1090 steel, underneath the notch root, following a diffuse notch surface instability [109] as verified in this the-
sis. But there have been other observations as will be discussed subsequently where hydrogen induced void initiation and growth occurs at a much earlier stage prior to link up. It is quite possible that since Garber et al used unnotched specimens, substantial straining, close to fracture, may have been necessary to induce plastic instability and substantial void initiation in these materials, so that the fast link up fracture which occurred may have masked any effect of hydrogen on plastic instability.

Cialone and Asaro [107] studied the effect of hydrogen precharging on fracture of smooth and notched cylindrical specimens of spheroidized 1015, 1017 and 1045 steels. They observed that hydrogen induced ductility loss by promoting both void initiation and growth. These voids grew primarily along grain and subgrain boundaries linking the carbides, prompting them to suggest that hydrogen induced decohesion brings about void initiation and growth at earlier stresses and strains. They observed that nonmetallic inclusions like sulfides brought about a change of fracture mode, from ductile rupture to quasicleavage, by absorbing hydrogen during charging and releasing it during deformation thus creating a dynamic charging [70] condition. It is worthwhile to note here that strain localization acting in conjunction with hydrogen could also have brought about earlier void initiation as was found by Lee et al [70]
Lee et al. [108],[70] studied the deformation and fracture of uncharged and charged u-notch bend specimens of spheroidized 1090 steel. They noted that void initiation and growth occurred along characteristic slip traces under the notch root for both cases, but occurred earlier in the presence of hydrogen. They suggested that the primary effect of hydrogen is to promote the onset of plastic instability. The instability process itself would then promote localized shear along the characteristic slip traces. This would give rise to elastic and plastic incompatibility stresses at the carbide particles intersecting the bands thus promoting decohesion. This decohesion then serves to help the surface cracks grow inwards from the notch to the bulk. But their work does not explain whether instability precedes void initiation and growth or occurs as a result of it. It is also not clear whether hydrogen promotes only instability or whether it also promotes void initiation and growth to a significant degree.

Onyewuenyi et al. [109] have shown that hydrogen promotes surface instability in spheroidized 1090 steel. This instability then gives rise to shear localization which promotes cracking along the characteristic slip traces. But events at the start of surface instabilities as well as the partitioning of hydrogen effects between instability and void profusion need further clarification.
Lin et al [111] studied hydrogen effects on shear localization in spheroidized 1009 and 1095 steels. They concluded that the effect of hydrogen was to impede shear localization at low and medium hydrogen input fugacities but enhance it at higher fugacities. They attribute this enhancement of instability to increased void growth caused by high fugacity hydrogen accumulating and generating pressure in preexisting voids. But prior to the onset of shear localization, there was no discernible effect of hydrogen on void initiation and growth, as was also reported by Lee et al [70] and Onyewuenyi et al [109]. Void initiation and growth does indeed accentuate and intensify the instability process as observed by Yamanoto [24], but whether void initiation and growth would precede instability in these systems and start it off is an open question. Moreover, Lin et al looked at the lateral surface near the notch root of their specimens, so that their observations pertain to plane stress deformation rather than plane strain deformation at the notch root.

Hydrogen induced void initiation and growth would constitute "irreversible damage" as has been found for iron [133]. But let us consider the following observations. A hydrogen induced decrease in lattice friction stress has been found with no indication of damage [112]. Increases in creep rate have been found in iron charged with low fugaci-
ty hydrogen before any significant void formation. There is evidence of reversible softening in spheroidized 1090 steel precharged with medium and high input fugacity hydrogen (<100A/m²) for less than 10 hours [85],[115]. But higher fugacities (150 A/m²) of hydrogen or longer charging times (>10 hrs.) cause irreversible damage in the form of voids and blisters and the hardness is not recovered on prolonged degassing. These results indicate that void and blister formation is not necessary for hydrogen induced softening or damage, the latter becoming important only at very high input fugacities or long charging times.

The foregoing treatment was on the effect of hydrogen on the so called "strain controlled fracture" of steels. But a number of medium and high strength steels have been reported to exhibit "stress controlled fracture" in the presence of hydrogen, which includes increasing amounts of intergranular and transgranular cleavage and mixed modes of intergranular and ductile rupture.

Lee et al [116] have studied the deformation and fracture of u-notched bend specimens of uncharged and charged 4340 steel. They found that hydrogen promoted mode I cracking in a region of internal stress concentration within the notch root plastic zone in contrast to their results on spheroidized 1090 steel. This mode I crack was connected to the surface by a mode II crack. The fracture mode in
the mode II region was dimple rupture and intergranular in mode I with increasing amounts of dimple rupture in the final stages of fracture. Their results are consistent with the model proposed by Troiano [66] of the need for a critical combination of stress and hydrogen concentration for early fracture in these steels. They also suggested that plastic instability plays an additive role in promoting fracture in these systems.

McMahon and coworkers [117]-[119], [120],[124] have studied the fracture of high strength steels in gaseous hydrogen atmospheres. Banerji et al [117] studied hydrogen induced fracture of quenched and tempered 4340 steels over a range of tempering temperatures. They found that the critical stress intensity for cracking in hydrogen atmospheres $K_{th}$ was reduced by a factor of five from that in air, when tempered in the martensitic embrittlement range. They concluded that this effect was due to P and N segregation at the grain boundary, presumably during the austenitizing treatment. The fracture was mostly intergranular in nature. Low Mn and Si contents favored mixed rupture and cleavage fracture with very little lowering of $K_{th}$. Addition of Mn and Si promoted intergranular fracture and reduced $K_{th}$ by a factor of five, due to enhancement of segregation at grain boundaries by Mn and Si.
Takeda et al [118] studied hydrogen induced fracture in 5% Ni steel. They found that in steels containing low Mn and Si (.02-.03 wt.%), hydrogen induced cracking occurred along two bands of plastic shear connecting the precrack. The resulting cracks were very intense and sharp as opposed to the blunt cracks observed in the uncharged steels. As Mn and Si was increased in the steels, the cracking became increasingly intergranular with cracking occurring internally between the surface and the site of maximum stress as was also observed by Lee et al [116].

Kameda et al [119] also observed hydrogen induced crack extension by mode II shear bifurcation in 3.5 Ni 1.7 Cr steels. But when doped with P, Sn or Sb, the $K_{th}$ decreased and the fracture became totally intergranular.

Bandyopadhyay et al [120] have studied the effects of composition, yield strength and hydrogen pressure on hydrogen induced cracking in 4340 steel. They observed that when yield strength and the hydrogen pressure were increased, the $K_{th}$ decreased and the extent of intergranular fracture also increased. Such an effect of yield strength has also been modelled by Gerberich [94] using a crack tip stress assisted hydrogen accumulation model. The Mn and Si induced segregation of P and S also drastically reduced $K_{th}$ and increased intergranular fracture.
All the aforementioned results have been explained using the Troiano model of decohesion which involves the effect of hydrogen accumulating in the maximum triaxial stress region under a crack. Furthermore, impurity weakened boundaries provide an easy path for hydrogen induced decohesion and fracture. But Kameda et al [121] have subsequently shown that the equilibrium hydrogen concentration in the region of maximum stress is only about $10^{-6}$ atom fraction, which is too small to explain the observed decohesion effect. So they proposed a dynamic interaction between hydrogen and a moving crack. As the crack moves, it carries its hydrogen atmosphere along aided by stress assisted diffusion of its hydrogen atmosphere. This atmosphere increases with time and enhances the decohesion of the boundary in front of the crack and thus enables the crack to accelerate. This model, however, will not work when the crack breaks away from its atmosphere at high velocities. Also in relatively pure steels, which fail by transgranular cleavage and ductile rupture this brittle cracking model will not work. In such steels, cracking occurs along lath boundaries [122] and translath slip planes, where dislocation assisted hydrogen transport along slip planes in laths [123] and subsequent hydrogen accumulation in translath slip planes and lath boundaries become more important.
Costa et al [104] studied hydrogen induced fracture of tensile and bend specimens of quenched and tempered 1045 steels tempered over a range of \(275^\circ C\) to \(400^\circ C\).

The uncharged tensile specimens showed a mixed fracture mode of intergranular (IG), quasicleavage (QC) and microvoid coalescence (MVC), with the latter two modes increasing with the tempering temperature. Introduction of hydrogen changed the fracture mode predominantly to IG fracture with QC and MVC fracture increasing slightly with tempering temperature. In the uncharged bend specimens, cracking originated at the notch root. Cracking was mode I in nature in contrast to the work of Lee et al [116] and was predominantly IG. Introduction of hydrogen caused subsurface mode I crack initiation with a mixture of IG and transgranular QC fracture. This QC fracture was across martensite lath packets, indicating that maybe hydrogen initiated QC fracture first. It is difficult to deduce from their results whether hydrogen effects are additive to those causing tempered martensitic embrittlement or not.

After reviewing the different results in the literature, an attempt can be made to rationalize and unify the diverse observations as has been done by McMahon [124]. Most of the results point towards two basic effects of hydrogen in steel. Hydrogen can promote localized shear by increasing screw dislocation mobility and decreasing cross slip. Then
this localized deformation can promote rupture at interfaces of inclusions and second phase particles. In an impure material containing group v elements, hydrogen acting in conjunction with segregated impurities can promote grain boundary decohesion and intergranular fracture.
Chapter II
EXPERIMENTAL METHODS

2.1 MATERIALS STUDIED

The materials studied include an AISI 1090 steel similar to the one used by Chang [128] and Onyewuenyi [85] and an AISI 1045 steel. The compositions of these steels are given in Table 1. The 1045 steel was received in the form of 24 mm square hot rolled bars and the 1090 steel was received in the form of 38 mm x 13 mm hot rolled plates.

Table 1
Composition of AISI 1045 and 1090 steels

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>AISI 1045</td>
<td>0.44</td>
<td>0.77</td>
<td>0.22</td>
<td>0.027</td>
<td>0.014</td>
</tr>
<tr>
<td>AISI 1090</td>
<td>0.88</td>
<td>0.75</td>
<td>0.19</td>
<td>0.036</td>
<td>0.017</td>
</tr>
</tbody>
</table>
2.2 HEAT TREATMENT AND MACHINING

The spheroidization of the 1090 steel was carried out by heating at $750^\circ C$ for two hours, furnace cooling to $704^\circ C$, holding at that temperature for twenty hours and then air cooling to room temperature. They were then finish machined to the dimensions shown in Figure 6. The notch root radius was 1.19 mm. Quenched and tempered microstructures were obtained as follows. The 1090 steel was rough machined, austenitized at $865^\circ C$ for half an hour and quenched in oil. Half the number of 1090 specimens were tempered at $288^\circ C$ for one hour and these will be referred to as 1090A steels. The other half were tempered at $385^\circ C$ for one hour and these will be referred to as 1090B steels. The 1045 steel was austenitized at $845^\circ C$ for one and a half hours and then quenched in iced water containing 5% polyethylene glycol. Half the number of specimens were tempered at $288^\circ C$ and these will be referred to as 1045A steels and the other half were tempered at $385^\circ C$ and these will be referred to as 1045B steels. Both the 1045 and the 1090 steels were then finish machined to the final dimensions shown in Figure 7. The notch root radius of the quenched and tempered 1090 steels was 1.5 mm and that of the 1045 steels was 1.19 mm. The typical microstructure of spheroidized 1090 steel is shown in Figure 8. The typical quenched and tempered...
microstructures of 1045 and 1090 steel are shown in Figures 9-12.
Figure 6: Dimensions of the 1090 specimen used in four point bending.
Figure 7: Dimensions of the quenched and tempered 1045 and 1090 steel
Figure 8: The microstructure of spheroidized 1090 steel.

Figure 9: The microstructure of 1045A steel.
Figure 10: The microstructure of 1045B steel.

Figure 11: The microstructure of 1090A steel.
Figure 12: The microstructure of 1090B steel.

2.3 SPECIMEN PREPARATION

The notch surfaces of all bend testing specimens were polished successively through 240, 320, 400 and 600 grit silicon carbide abrasive paper and with diamond paste from 6 microns to 1 micron. The direction of the final polishing was parallel to the direction of the maximum principal stress. This was done to remove machining marks and the accompanying deformed layer on the surface, and to provide a standard surface for all experiments smooth enough for scanning electron microscopic observations.
2.4 UNCHARGED BEND TESTS.

All bend tests were carried out on an Instron machine with a 5000 Kgf capacity GRM compression/tension load cell. The cross head speed that was employed was .05 cm/min. This corresponded to a strain rate of $1.33 \times 10^{-3}/s^{-1}$ for plane strain tension tests and a maximum surface strain rate of $7.3 \times 10^{-4}/s^{-1}$ for bend tests.

2.4.1 Four Point Bend Tests

The spheroidized 1090 steel specimens were subjected to four point bending. This enabled the study of both the tensile and compressive modes of deformation at the notch root. The four point bending fixture in both the tensile and compressive mode is shown in figures 13 and 14 respectively.

The notch root strain which remains essentially constant over a region of $0.6p$ ($p$=notch root radius) across the notch midplane, was measured by monitoring the displacement of two parallel scribe lines on the notch surface with increasing load. The notch opening strain was measured by monitoring the notch edge displacement as shown in figure 15. This strain was correlated with the notch root strain. This procedure enabled calculation of the notch root strain on hydrogen precharged notches as discussed later. The notch surface was viewed under the SEM and the notch deformation patterns studied as a function of notch root strain.
Figure 13: The four point bending fixture in tension.

Figure 14: The four point bending fixture in compression.
The notch surface rumpling was measured using interference microscopy by producing optical interference patterns of the notch root surface. Monochromatic thallium light was used to produce fringes and the notch surface roughness was calculated from the deflection of the fringes. The notch surface roughness was recorded as a half amplitude of the rumpling on the notch surface.

![Diagram of notch opening displacement](image)

**Figure 15:** Measurement of notch edge displacement.

### 2.4.2 Three point bend testing

The quenched and tempered 1045 and 1090 steels were subjected to three point bending. The three point bend testing fixture is shown in Figure 16. The notch root strain and the notch opening strain were monitored with load in the
same manner as in four point bending. The notch surface was observed under the SEM as mentioned in the previous section.

Figure 16: Three point bending fixture

2.4.3 Plane Strain Tension and Compression Testing

Spheroidized 1090, quenched and tempered 1090 and 1045 steels were all subjected to plane strain tension in the Instron machine using a cross head speed of .05 cm/min. The dimensions of the plane strain tension specimen is shown in figure 17. The strain was measured using a COD gage between two knife edges welded to the gage length of
the sample. The plane strain true stress-true strain curve was then plotted.

![Figure 17: Dimensions of the plane strain tension specimen](image)

Plane strain compression testing was carried out on spheroidized 1090 steel. The compression specimen was a sheet with the dimensions as shown in Figure 18. This sheet was pressed between two hardened 1090 steel dies with the same cross head speed as mentioned above. The dies were 2.71 mm thick and the compression fixture looks as shown in figure 19. Teflon and grease were used as lubricants between the die and the specimen during the test. The strain was measured by monitoring the thickness change
of the sheet as a function of load. The true stress-true strain curve in plane strain compression was then plotted.

Figure 18: Dimensions of the plane strain compression specimen.
2.4.4 Tests with Hydrogen Charging

Two nichrome wires were welded to the specimen which was then coated with nonconducting acid resistant paint on all surfaces except the notch surface which was to be charged. The electrolyte used for hydrogen charging was $1\text{M} \ \text{H}_2\text{SO}_4$ with 1g/liter of thiourea as a hydrogen recombination poison. The solution was bubbled with pure nitrogen for at least 3 hours prior to charging to reduce the dissolved oxygen content in the solution. Purification
of the nitrogen gas was achieved by passing it through three gas washing bottles of chromous solutions, which removes the oxygen from the gas stream.

The anode in the cell was a fine platinum wire which was placed in the charging cell close to the notch root of the specimen. The charging cell configuration is shown in Figure 20. An Aardvark potentiostat/galvanostat model V-2LR was used to provide the galvanostatic charging current.

The specimen was put in the charging cell and the potentiostat turned on. The electrolyte was then introduced into the cell which was bubbled with purified nitrogen throughout the duration of charging of two hours. The charging currents that were imposed are:

1. 100 A/m² for the spheroidized 1090 steel.
2. 15 A/m² for the 1045 and 1090 quenched and tempered steels (both A and B).
3. 80A/m² for the 1045 and 1090 quenched and tempered steels (both A and B).

After charging, the specimen was removed and washed in water, stripped of the microstop, rinsed in acetone and blow dried. The specimen was mounted in the loading fixture and the load was applied within two minutes of removal from the cell. The spheroidized 1090 steel was subjected to four point bending with the notch in tension or compression as the case maybe. The quenched and tempered 1045 and
Figure 20: Charging cell used for galvanostatic hydrogen charging.
1090 steels were subjected to three point bending with the notch in tension. The notch opening strain was measured as before. The notch root strain could not be measured directly in the case of these hydrogenated samples because scribes were not made on the notch surface. The scribes, if put on the surface, might have served as sites for hydrogen induced cracks and thus vitiated the results. So the measured notch opening strain was used to obtain the notch root strain with the help of the corellation obtained between notch opening strain and the notch root strain in the uncharged bend tests. The notch surface was studied as before under the scanning electron microscope. Since the notch surface had lost its optical finish, interference microscopy was ineffective in studying surface rumpling. So the notch root profile obtained after notch midplane sectioning was observed and surface rumpling data was obtained from it.

2.5 MICROSCOPY
As was mentioned before, the notch surfaces of both charged and uncharged specimens were studied under the SEM to observe the notch deformation behavior. After this was done, the notch was sectioned at its midplane. This midplane section was then polished to a 1 micron finish and etched with 4% picral and 5% nital. The notch root and the
underlying section were then observed using the scanning electron microscope to study cracking and void formation. Energy dispersive analysis of X-rays (EDAX) was used to identify inclusions.
Chapter III

RESULTS

3.1 EMPirical STRAIN RELATIONS

The following empirical relations between engineering notch root strain \( e_n \) and engineering notch edge opening strain \( e_e \) were obtained in the uncharged specimens.

For spheroidized 1090 steel, with the u-notch in tension,

1. \( e_n = 4.234e_e + 0.0016 \) for \( e_e < 0.014 \)

2. \( e_n = 0.957e_e + 0.032 \) for \( e_e > 0.014 \)

For spheroidized 1090 steel, with the u-notch in compression,

1. \( e_n = 1.095e_e - 0.007 \) for \( e_e < 0.089 \)

2. \( e_n = 0.895e_e + 0.008 \) for \( e_e > 0.089 \)

For 1045A steel,

1. \( e_n = 1.178e_e + 0.007 \)

For 1045B steel,

1. \( e_n = 1.457e_e - 0.0022 \)

For 1090A steel,

1. \( e_n = 0.816e_e + 0.01 \)

For 1090B steel,

1. \( e_n = 0.783e_e + 0.0075 \)
3.2 MECHANICAL PROPERTIES OF 1045 AND 1090 STEELS

The data on the mechanical properties obtained in plane strain tension or compression are tabulated in Table 2.

Table 2

<table>
<thead>
<tr>
<th></th>
<th>Spheroidized 1090</th>
<th>Quenched and Tempered 1045</th>
<th>Quenched and Tempered 1090</th>
</tr>
</thead>
<tbody>
<tr>
<td>U-notch in Tension</td>
<td>298</td>
<td>1551</td>
<td>882</td>
</tr>
<tr>
<td>U-notch in Compn.</td>
<td>302</td>
<td>1697</td>
<td>1711</td>
</tr>
<tr>
<td>Yield Strength (MPA)</td>
<td></td>
<td>1760</td>
<td>1588</td>
</tr>
<tr>
<td>U.T.S (MPA)</td>
<td>765</td>
<td>1697</td>
<td>1711</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1760</td>
<td>1588</td>
</tr>
<tr>
<td>$\varepsilon_u$</td>
<td>0.143</td>
<td>0.017</td>
<td>0.054</td>
</tr>
<tr>
<td>R. A.%</td>
<td>31</td>
<td>1.7</td>
<td>34.3</td>
</tr>
</tbody>
</table>
3.3 SLIP BANDS, SURFACE RUMPLING AND FRACTURE IN SPHEROIDIZED 1090 STEEL

3.3.1 Uncharged Specimens

Micrographs I(a)-I(e) in Plate I show the evolution of slip bands on the u-notch surfaces in tension with increasing notch root strain. As the strain is increased, the slip lines increase in number and density and the surface loses its shiny appearance and becomes rough. The slip lines are wavy owing to the polycrystallinity of the material. At a true strain of about 0.264, the slip lines have grouped together in bands running perpendicular to the loading direction and across a few grain diameters as seen in micrograph I(b). On further straining, these deformation bands become more intense. This strain is then characterized as the strain for the onset of surface instability. Microvoids start forming at low strains but these are few in number and do not seem to be connected with the deformation bands. These microvoids are usually formed by either breaking or decohesion of carbide particles. But at strains beyond 0.264, these voids, when they intersect the deformation bands, become influential in promoting the growth of microcracks as can be seen in micrographs I(d)-I(e) in Plate I. This process thus promotes the growth of surface instability. The micrographs in plate Plate II show the rapid growth of surface microcracks in
these slip bands at a strain of 0.672 which is then charac-
terized as the strain for surface microcracking.

A better idea of the surface roughening during surface
instability is obtained by consideration of the evolution
of surface rumpling with strain as shown in Figure 21. There is an increase in the rate of growth of the surface
waves at a strain of 0.21 which is in excellent agreement
with the strain for instability of 0.264 obtained by obser-
vation of the notch surface. Micrograph III(a) in plate
Plate III shows the extent of surface rumpling at a strain
of about 0.457. In micrograph III(b) the intense surface
rumpling has given rise to the growth of shear bands and
voids underneath the notch root. It is noteworthy that
this shear band has not propagated very far underneath the
notch root even at this large strain.

The evolution of slip lines and bands on the notch sur-
face with the u-notch in compression is shown in micro-
graphs IV(a)-IV(e) in Plate IV. It is interesting to note
that the slip lines are not as wavy as those in tension and
tend to be planar in nature. Slip bands become prominent
at a strain of 0.15 and these then grow rapidly with fur-
ther strain. The absence of voids on the surface is not
surprising. Cracks start forming in these slip bands, due
to the growth of these surface instabilities, at a strain
of 0.405 and then rapidly grow in width and length as seen
Plate I

Slip bands on u-notch surface in uncharged sph 1090 in tension

(a) ε = 0.207 (b) ε = 0.264 (c) ε = 0.35 (d) ε = 0.473 (e) ε = 0.577
Plate II

Slip bands on u-notch surface in uncharged sph 1090 in tension

(a) $\varepsilon = 0.672$  (b) $\varepsilon = 0.79$
Figure 21: Evolution of notch surface rumpling as a function of strain
Plate III

Surface rumpling in tension in uncharged aph 1090 steel

(a) $\varepsilon = 0.457$

(b) $\varepsilon = 0.79$
in micrographs V(a)-V(c) in Plate V. Surface waves, as observed in figure 21, grow rapidly beyond a strain of about 0.15, signifying the onset of surface instability in excellent agreement with notch surface observations. It is interesting to note that the amount of roughening in compression is much higher than that in tension. Micrograph VI(a) in Plate VI shows the intense surface rumpling at a strain of 0.38 and micrograph 6(b)-6(c) show the growth of cracks underneath the notch root as a result of intense shearing at the bottom of the surface waves. It is worthwhile to note here that in these samples loaded in compression, some residual tensile stresses at the notch surface would result due to elastic springback of the undeformed material near the notch. But the morphology of the cracks suggest that they have opened up as a result of shearing under the notch root under mixed mode I-mode II loading and not due to pure tensile mode I loading. Also no voids were observed on the notch surface and voids that were found under the notch surface have mostly formed ahead of the crack front. Finally, because of plastic relaxation on initial loading, the bending moment is reduced and the reverse bending moment on unloading is reduced. Thus, the residual tensile stress at the notch root on unloading should be much less than the compressive stress during loading mitigating against any cracking induced by the
residual tensile stresses. These arguments suggest that the residual tensile stresses at the notch surface do not contribute significantly to void and crack formation at the notch surface under compressive loading. It will be interesting to compare these experimental observations with the continuum mechanics predictions and this is done in the discussion section.

The fracture surface of uncharged spheroidized 1090 steel is shown in Plate VII. Near the notch root the fractograph shows shear dimples indicating the extensive shear that has occurred there. Deeper into the material, the fracture mode changes to equiaxed dimple rupture near the high triaxial stress area underneath the notch, indicating that fracture occurred there by void growth and coalescence. A little deeper into the bulk, this mode changes to mostly transgranular cleavage with some intergranular cracking, which is indicative of unstable fracture.
Plate IV
Slip bands on u-notch surface in uncharged sph 1090 in compression

(a) $\varepsilon = 0.113$ (b) $\varepsilon = 0.15$ (c) $\varepsilon = 0.17$ (d) $\varepsilon = 0.244$ (e) $\varepsilon = 0.3$
Plate V

Slip bands on u-notch surface in uncharged sph 10%0 in compression

(a) \( \epsilon = 0.41 \) (b) \( \epsilon = 0.51 \)
Plate VI

Surface rumpling in compression in uncharged sph 1090 steel

(a) $\epsilon = 0.585$ (b) $\epsilon = 0.565$ (c) $\epsilon = 0.565$
Plate VII

Fracture surface of uncharged spheroidized 1090 steel.

(a) Shear dimples near the notch root (b) Equiaxed dimples a little deeper into the material (c) Cleavage fracture in the bulk.
3.3.2 Precharged Specimens

Micrographs VIII(a)-VIII(d) in Plate VIII show the evolution of slip bands in tension. Slip bands are observed at a strain of 0.135 and grow rapidly with increasing strain. In a high contrast picture as in micrograph VIII(b), the intense surface rumpling is observed easily. Microvoids, which form at low strains, are not very influential until a strain of 0.282 or more is reached. Then they start to promote microcackling along the slip bands, as will be seen subsequently. Microcracks start to develop on the surface at a strain of about 0.31. The voids, usually formed due to carbide cracking or decohesion, seem to promote the growth of microcracks as seen in micrograph VIII(c). At a higher strain of 0.424, the cracks increase in width and extent under the applied tensile stresses. Secondary cracks also form at the notch surface and these tend to link up with the primary crack as observed in micrograph VIII(d). The rumpling data as shown in Figure 21 indicate that there is rumpling occurring much earlier in the precharged specimen as compared to the uncharged specimens. Micrograph IX(a) in Plate IX shows the growth of the crack into the material as it follows the characteristic slip traces. A closer observation of the crack root, micrograph IX(b), reveals that cracking occurs by void initiation and growth ahead of it. These voids form at carbide interfaces.
and grain boundaries which lie on the characteristic slip traces.

The fracture surface is shown in micrographs X(a)-X(c) in Plate X and three distinct zones are observable. In the first zone which is closest to the notch root, the fracture mode is a mix of quasicleavage and dimple rupture and is associated with ductile tearing as the crack proceeded inwards from the notch surface. This then changed to an equiaxed dimple rupture around the triaxial stress region and then to subsequent unstable fracture characterized by intergranular and transgranular cleavage.

Micrographs XI(a)-XI(d) in Plate XI show the evolution of slip lines on the notch surface in precharged specimens in compression. Here a surface instability starts at a strain of about 0.125 and grows rapidly with increasing strain. At a strain of 0.375, cracks start to develop and grow along the bottom of the slip bands. No significant voids on the notch surface were observed, like in the uncharged specimens. The surface rumpling data in Figure 21 show no significant difference from those of the uncharged specimens in compression. The onset of the rapid growth of surface waves is in agreement with the observations on the u-notch surface. Micrograph XII(a) in Plate XII shows the intense rumpling on the notch surface and cracks underneath the notch emanating from the bottom of the surface waves.
Plate VIII

Slip bands on u-notch surface in precharged sph 1040 in tension

(a) ε = 0.135 (b) ε = 0.21 (c) ε = 0.305 (d) ε = 0.424
Cracking at notch root in precharged sph. 1090 steel in tension

Plate IX

(a) Cracking below the notch at \( \varepsilon = 0.424 \).

(b) Crack propagation by void nucleation and growth.
\[ \varepsilon = 0.424 \]
Plate X
Fracture surface of precharged 1090 steel

(a) Near the notch root (b) A little deeper into the material underneath the notch (c) In the bulk of the material.
At a higher strain of 0.545, the notch profile reveals cracking underneath the surface instabilities (micrograph 12(b)). It is interesting to note that voids have initiated underneath the notch root in spite of the compressive stresses acting here (micrograph 12(c)). These voids are entirely due to the plastic and elastic incompatibility stresses generated at the carbide interfaces due to the extensive shearing action in this region. In uncharged specimens in compression, at a comparable strain, the degree of void profusion is much less (micrograph VI(c)). This suggests that hydrogen does have a pronounced effect in promoting void formation through its effect on the cohesive strength at interfaces as will be discussed later. The observations in this subsection are summarized in Table 3.
Plate XI

Slip bands on u-notch surface in precharged sph 1090 in compn.

(a) $\varepsilon = 0.125$ (b) $\varepsilon = 0.285$ (c) $\varepsilon = 0.375$ (d) $\varepsilon = 0.502$
Plate XII

Surface rumpling in compression in precharged sph 1090 steel.

(a) Rumpling and cracking at the notch root due to shear; $\epsilon = 0.545$

(b)Cracking and void formation under the notch; $\epsilon = 0.545$
Table 3

Critical strains in sph 1090 steel.

<table>
<thead>
<tr>
<th></th>
<th>Spheroidized 1090 Uncharged</th>
<th>Spheroidized 1090 Precharged at 100A/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>U-notch in Tension</td>
<td>U-notch in Compression</td>
</tr>
<tr>
<td>Surface Instability Strain</td>
<td>0.264</td>
<td>0.15</td>
</tr>
<tr>
<td>Surface Rumpling Strain</td>
<td>0.21</td>
<td>0.15</td>
</tr>
<tr>
<td>Crack Initiation strain</td>
<td>0.672</td>
<td>0.405</td>
</tr>
<tr>
<td>Load at Crack Initiation</td>
<td>17.66</td>
<td>13.73</td>
</tr>
</tbody>
</table>

3.4 SLIP BANDS AND FRACTURE IN QUENCHED AND TEMPERED STEEL

3.4.1 Uncharged Specimens

Evolution of slip bands in 1045A steel is shown in micrographs XIII(a)-XIII(d) in Plate XIII. Interestingly, the slip bands are very sharp and intense and seem to contain a lesser number of slip lines as compared to the spheroidized 1090 steel. The slip bands form at a very low strain of 0.026 and increase in length and intensity with increasing strain. An interesting feature is the formation of micro-
cracks at low strains of 0.026 in the slip bands. The specimen fractured at a strain of 0.177 at a load of 30.9 kN, by the growth of these microcracks into the bulk. The fracture surface shows a dimpled shear mode near the notch root which then changes to a predominantly intergranular cleavage mode as seen in micrographs XIV(a)-XIV(b) in Plate XIV.

In the uncharged 1045B steel, surface instability occurs at a strain of 0.115 as can be seen from micrographs XV(a)-XV(c) in Plate XV. At this strain, intense microcracking was also observed as shown in micrograph 15(b). The growth of these microcracks becomes pronounced at a strain of about 0.26. The specimen fractured at a strain of 0.31 at a load of 23.6 kN. The fracture surface shows a shear dimple mode near the notch root which changes to mostly microvoid coalescence as seen in micrograph XVI(a)-XVI(b) in Plate XVI. There is much more dimple rupture in this region of fracture in the 1045B steel than in the 1045A steel. The surface profile shows intense rumpling at a strain of 0.26 and a closer examination reveals hole growth by intense shearing at the bottom of the surface wave as observed in micrograph XVII(a)-XVII(b) in Plate XVII.
Plate XIII

Slip bands on the u-notch surface of uncharged 1045A steel

(a) $\varepsilon = 0.026$ (b) $\varepsilon = 0.037$ (c) $\varepsilon = 0.093$ (d) $\varepsilon = 0.177$
Plate XIV

Fracture surface of uncharged 1045A steel.

(a) Mode II shear fracture near the notch root.
(b) IG and TG cleavage fracture in the bulk.
Plate XVI

Fracture surface of uncharged 1045B steel.

(a) Mode II shear fracture near the notch root
(b) Fine dimple fracture in the bulk.
Plate XVII

Surface rumpling in uncharged 1045B steel.

(a) Rumpling at the notch root; \( \varepsilon = 0.204 \)

(b) Voids formed by shear at the bottom of the surface wave; \( \varepsilon = 0.204 \)
3.4.2 Precharged Specimens (Charging Current Density 15A/m²)

Long cracks on the surface were observed at a strain of 0.013 and there was not much in the way of surface instability at this strain in 1045A steels as seen in micrograph XVIII(a) in Plate XVIII. The notch root profile at this strain showed mainly mode I intergranular cracking which got wider as it went deeper into the notch as is evident in micrograph VIII(b). Fracture occurred at a strain of 0.071 at a load of 13.5 kN. At this strain, bands of intense instability had developed on the surface (micrograph VIII(c)). The fracture surface showed a mode II shear fracture region near the notch root, which changed to mostly intergranular fracture with limited dimple rupture in the interior as shown in micrographs XIX(a)-XIX(b) in Plate XIX.

At low strains, the 1045B steel did not show well developed slip bands either, but there were pockets of highly localized deformation at a strain of 0.023 as seen in micrograph XX(a) in Plate XX. Microcracks were observed in the pockets as well as in other areas, presumably austenite grain boundaries. At higher strains there were more pockets of localized deformation and increasing microcracking (micrograph XX(b) and (c)). Fracture occurred at a strain of 0.073 at a load of 15.1 kN. A u-notch profile showed mode
Plate XVIII

Notch surface and notch profile of precharged 1045A steel.

(a) $\varepsilon = 0.013$  (b) $\varepsilon = 0.013$  (c) $\varepsilon = 0.059$
Plate II
Fracture surface of precharged 1045A steel

(a)

(b)

(a) Mode II shear fracture near the notch root.

(b) TG, TG and MVC fracture in the bulk.
II cracking near the notch surface up to a depth of 0.96 mm and a mode I intergranular crack from thereon (micrograph XX(d)). The fracture surface showed mode II quasicleavage tearing which changed to mostly dimpled rupture with some intergranular cracking (micrograph XXI(a)-XXI(b) in Plate XXI). Like in the uncharged case, here also more ductile rupture was observed than in the case of 1045A steel.
Plate XX

Notch surface and notch profile of precharged 1045 steel

(a) $\varepsilon = 0.023$  (b) $\varepsilon = 0.058$  (c) $\varepsilon = 0.073$  (d) $\varepsilon = 0.073$
Plate XXI

Fracture surface of precharged 1045B steel

(a) Mode II fracture near the notch root.
(b) Fine dimple fracture in the bulk.
3.4.3 Precharged Specimens (Charging Current Density 80A/m²)

At this charging current, the 1045A specimens showed crack initiation at a strain of 0.001 with very little surface instability as shown in micrograph XXII(a) in Plate XXII. The notch root profile showed mode I intergranular cracking (micrograph XXII(b)). At a higher strain of 0.015 some instability on the surface is observed and the notch root profile shows mode II cracking at the notch root followed by mode I cracking. Fracture occurred at a strain of 0.03 and at a load of 9 kN. The fracture surface showed a very small quasi-cleavage area near the notch root followed by mostly intergranular fracture (micrograph XXIII(a) (b) in Plate XXIII).

The 1045B steels showed microcracks at a strain of 0.023 with no attendant surface instability (micrograph XXIV(a)). At higher strains, a long mode II shear crack was observed running across the notch surface (micrograph XXIV(b) in Plate XXIV). A notch root profile revealed a long mode II crack about 0.26 mm in depth and subsequent mode II cracking (micrograph XXIV(c)). Fracture occurred at a load of 15.4 kN at a strain of 0.072. The fracture surface revealed a region of a mixed intergranular and quasi-cleavage fracture followed by mostly ductile rupture with very little intergranular cracking (micrograph XXV(a)-(c) in
Plate XXII

Notch surface and notch profile of precharged 1045A steel

(a) $\varepsilon = 0.001$ (b) $\varepsilon = 0.001$ (c) $\varepsilon = 0.015$ (d) $\varepsilon = 0.015$
Plate XXIII

Fracture surface of precharged 1045A steel.

(a) Mode II fracture near the notch root.
(b) IG and MVC fracture in the bulk.
Plate XXV). The foregoing observations are summarized in Table 4.
Plate XXIV

Notch surface and notch profile of precharged 1045 steel.

(a) \( \epsilon = 0.025 \)  (b) \( \epsilon = 0.038 \)  (c) \( \epsilon = 0.038 \)
Plate XXV

Fracture surface of precharged 1045B steel.

(a) Mode II fracture near the notch root.
(b) MVC and IG fracture in the bulk.
### Table 4

**Critical strains for quenched and tempered 1045 steel.**

<table>
<thead>
<tr>
<th></th>
<th>Uncharged</th>
<th>Precharged 15A/m²</th>
<th>Precharged 80A/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1045 A</td>
<td>1045 B</td>
<td>1045 A</td>
</tr>
<tr>
<td><strong>Surface Instability Strain</strong></td>
<td>0.026</td>
<td>0.115</td>
<td>-</td>
</tr>
<tr>
<td><strong>Surface Cracking Strain</strong></td>
<td>0.026</td>
<td>0.115</td>
<td>0.013</td>
</tr>
<tr>
<td><strong>Fracture Strain</strong></td>
<td>0.177</td>
<td>0.31</td>
<td>0.071</td>
</tr>
<tr>
<td><strong>Load at Fracture (K)</strong>*</td>
<td>30.9</td>
<td>23.6</td>
<td>13.5</td>
</tr>
</tbody>
</table>

### 3.5 SLIP BANDS AND FRACTURE IN QUENCHED AND TEMPERED STEEL.

#### 3.5.1 Uncharged specimens.

The 1090A steel showed isolated pockets of localized deformation with no well developed slip bands at low strains of 0.016. But microcracking at this strain, presumably along prior austenite boundaries, was already evident. At a strain of 0.037, well developed slip bands are seen and these slip bands contain extensive microcracks (micrograph XXVI(b) in Plate XXVI). The slip bands increase in length...
and extent with a concomitant increase in microcracking within them, on subsequent straining to a strain of 0.055 (micrograph XXVI(c)). The notch root profile revealed some rumpling but no cracks under the notch root. Fracture occurred suddenly at a strain of 0.057 at a load of 31.4 kN. The fracture surface revealed a mode II shear and quasicleavage region about 100 microns in width. This was followed by mostly intergranular cracking with some dimpled rupture (Plate XXVII).

The 1090B steel showed well developed slip bands at a strain of 0.03 with extensive microcracking within them (micrograph XXVIII(b) in Plate XXVIII). Microcracking had already developed at lower strains of 0.01 (micrograph XXVIII(a)). Fracture occurred suddenly at a strain of 0.054 at a load of 31.1 kN. The fracture surface showed a mode II shear and quasicleavage region near the notch root about 150 microns in width. This was followed by a mix of intergranular cracking and substantial dimple rupture (Plate XXIX).
Plate XXVI

Notch surface of uncharged 1090A steel

(a) \( \epsilon = 0.016 \) (b) \( \epsilon = 0.037 \) (c) \( \epsilon = 0.055 \)
Plate XXVII

Fracture surface of uncharged 1090A steel

(a) Mode II fracture near the notch root.

(b) IG and MVC fracture in the bulk.
Plate XXVIII

Notch surface of uncharged 1090B steel

(a) $\varepsilon = 0.01$  (b) $\varepsilon = 0.03$
Plate XXIX

Fracture surface of uncharged 1090B steel

(a) Mode II fracture near the notch root.
(b) IG and MVC fracture in the bulk.
3.5.2 Precharged Specimens (Charging Current Density 15A/m²).

In 1090A steel, a long crack had already developed at a strain of 0.011 with almost no slip lines on the surface (micrograph XXX(a) in Plate XXX). The notch root profile revealed a mode I intergranular crack with some discontinuous crack segments (micrograph XXX(b)). Fracture occurred at a strain of 0.018 at a load of about 4 kN. The fracture surface showed mostly intergranular fracture with more ductile rupture as the crack moved into the bulk (Plate XXXI). There was no mode II region on the crack surface in sharp contrast to the uncharged specimen.

In the 1090B specimen, crack initiation occurred at a strain of 0.0098 (micrograph XXXII(a) in Plate XXXII). Even though there were no slip lines on the surface at this strain, there was some localized deformation associated with the crack. The notch root profile revealed a wide mode I intergranular crack running through the bulk and connected by a small mode II crack at the surface (micrograph XXXII(b)). In some places the intergranular crack followed sulfide inclusions which presumably offered an easy crack path. At a slightly higher strain of 0.01, another specimen showed a longer crack on the surface (micrograph XXXII(c)). The notch root profile showed mostly mode I intergranular cracking (micrograph XXXII(d)).
Plate XXX

Notch surface and notch profile of precharged 1090A steel

(a) $\varepsilon = 0.011$ (b) $\varepsilon = 0.011$
Plate XXXI

Fracture surface of precharged 1090A specimen.
Fracture occurred at a strain of 0.031 at a load of 6.1 kN. The fracture surface revealed a mode II shear and quasi-cleavage region about 100 microns wide followed by a mix of intergranular and dimple rupture (Plate XXXIII).
Plate XXXII

Notch surface and notch profile of precharged 1090B steel

(a) $\varepsilon = 0.0098$  (b) $\varepsilon = 0.0098$  (c) $\varepsilon = 0.01$  (d) $\varepsilon = 0.01$
Plate XXXIII

Fracture surface of precharged 1090B steel

(a) Mode II fracture near the notch root.
(b) IG and MVC fracture in the bulk.
3.5.3 Precharged Specimens (Charging Current Density 80A/m²)

A long crack was observed in 1090A specimens at a strain of 0.015, with some localized plasticity associated with the crack. (micrograph XXXIV(a) in Plate XXXIV). The notch profile revealed a long mode I intergranular crack which intersected the MnS inclusions at some places (micrograph XXXVI(b)). Fracture occurred at a strain of 0.022 at a load of 3.7 kN. The fracture surface was predominantly intergranular near the notch root with small pockets of dimpled rupture (Plate XXXV). The amount of dimpled rupture increased as the crack moved into the bulk. Once again there was no mode II region near the notch root. One additional feature of interest was pockets of quasicleavage fracture near undissolved carbide inclusions. It is possible that the carbides absorb hydrogen during charging and release it during straining thus promoting quasicleavage fracture near it, as has been observed with sulfide inclusions in spheroidized steels [107].

In the 1090B specimens, a long crack was observed at a strain of 0.01, with some localized plasticity near the crack (micrograph XXXVI(a) in Plate XXXVI). The notch profile revealed a zigzag mode I intergranular crack which looked wider in the bulk of the specimen than at the notch root (micrograph XXXVI(b)). On loading to a higher strain
Plate XXXIV

Notch surface and notch profile of precharged 1090 A steel.

(a) $c = 0.015$ (b) $c = 0.015$
Plate XXXV

Fracture surface of precharged 1090A steel.
of 0.015, a longer crack was observed on the notch root (micrograph XXXVI(c)). The notch root profile revealed a very small mode II crack at the notch root followed by a zigzagging intergranular crack (micrograph XXXVI(d)). Fracture occurred at a strain of 0.021 at a load of 5.35 kN. The fracture surface consisted mostly of intergranular and dimple rupture with no noticeable mode II region near the notch root (Plate XXXVII). The results on the quenched and tempered 1090 steels are summarized in Table 5.
Plate XXXVI

Notch surface and notch profile of precharged 1090B steel.

(a) $e = 0.01$  (b) $e = 0.01$  (c) $e = 0.015$  (d) $e = 0.015$
Plate XXXVII

Fracture surface of precharged 1090B steel.
Table 5

Critical strains in 1090 steel

<table>
<thead>
<tr>
<th></th>
<th>Uncharged</th>
<th>Precharged 15A/m²</th>
<th>Precharged 80A/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1090 A</td>
<td>1090 B</td>
<td>1090 A</td>
</tr>
<tr>
<td>Surface Instability Strain</td>
<td>0.037</td>
<td>0.03</td>
<td>-</td>
</tr>
<tr>
<td>Surface Cracking Strain</td>
<td>0.016</td>
<td>0.01</td>
<td>0.011</td>
</tr>
<tr>
<td>Fracture Strain</td>
<td>0.057</td>
<td>0.054</td>
<td>0.018</td>
</tr>
<tr>
<td>Load at Fracture (Kn)</td>
<td>31.4</td>
<td>31.1</td>
<td>4.0</td>
</tr>
</tbody>
</table>
4.1 UNCHARGED SPECIMENS

4.1.1 Surface Instability

In the u-notched specimen, the middle portion of the notch undergoes plane strain deformation. Thus the strains for surface instability observed in these specimens can be compared with the theoretical model proposed by Hutchinson et al [19]. According to this model, bifurcation can occur when a vertex develops on the yield surface in principal stress space. The condition for surface bifurcation for a hyperelastic solid as mentioned in the literature survey section is reproduced here.

\[
\frac{\sigma_2}{4\mu^\#} = 1 + \frac{\sigma_2}{4} [(2\mu - \sigma_2)(2\mu + \sigma_2)]^{1/2}
\]

\[\mu = \frac{1}{3} [E_s(\epsilon_1 - \epsilon_2) \coth(\epsilon_1 - \epsilon_2)] \]

\[\mu^\# = \frac{1}{3} [E_s - (E_s - E_t)(\cos\alpha + 1/2 \sin2\alpha)(1 + \sin2\alpha/2)^{-1}] \]

The principal stresses at the surface \((X_1 = 0)\) are given by,

\[\sigma_1 = 0 \quad \text{(10)}\]

\[\sigma_2 = \frac{2}{3}[E_s(\cos\alpha + 2\sin\alpha)X] \quad \text{(11)}\]

\[\quad - 122 -\]
\[ \sigma_3 = \frac{2}{3} [E_s (\sin \alpha + 2 \cos \alpha)]X \]  

(12)

where \( X \) and \( \alpha \) are the magnitudes of the strains \( \varepsilon_2 \),
and \( \varepsilon_3 \).

\[ \varepsilon_2 = X \cos \alpha, \quad \varepsilon_3 = X \sin \alpha, \quad \varepsilon_1 = -\varepsilon_2 - \varepsilon_3 \]  

(13)

For the plane strain case considered here,
\[ \alpha = 0 \text{ or } \pi \]  

(14)

Hence \( \varepsilon_3 = 0 \) and \( \varepsilon_1 - \varepsilon_2 = 2 \varepsilon_2 \)

\[ \sigma_2 = \frac{4}{3} E_s \varepsilon_2 = E_{sp} \varepsilon_2, \quad \frac{4}{3} E_t = E_{tp} \]  

(15)

where \( E_{sp} \) is the plane strain secant modulus and \( E_{tp} \) is the plane strain tangent modulus. Substituting
Equation 4-Equation 16 into Equation 3 gives,
\[ \frac{E_t}{E_s} = \frac{E_{tp}}{E_{sp}} = \frac{\varepsilon^*}{2} \left[ 1 - \exp\left(-2\varepsilon^*\right) \right] \]  

(17)

This equation relates the local strain hardening parameter \( E_{tp}/E_{sp} \) with the critical strain at instability in tension or compression. To compare this model with the experimental results, the moduli \( E_{sp} \) and \( E_{tp} \) have to be determined. These moduli can be obtained from a true stress-true strain plot of the material tested in plane strain condition. This can be done because the notch root behaves like a small plane strain tension specimen [129] and [27]. The plane strain tension and compression curves are shown in Figures 21 and 22. The ratio \( E_{tp}/E_{sp} \) for tension is calculated at a strain of 0.264 from the
tension curve and at 0.15 for compression from the compression curve. These $E_{tp}/E_{sp}$ values when inserted into Equation 17 yield strains of 0.45 and 0.26 for tension and compression respectively. These values differ somewhat from the experimental values, but the disagreement is of the same order as that found by Anand et al [110] in strains for the onset of shear bands in maraging steel. These theoretical strains at least agree well with the trend of the experimental observations which indicate that surface instability occurs earlier in compression than in tension. The disparity between experiment and theory could be due to additional effects like nonnormality and kinematic hardening not considered in the theory. The observed growth of surface instabilities (Figure 4) follows the theoretical predictions quite well, but the rumpling is much more enhanced in compression than in tension.

The vertex model does predict an earlier instability in compression. Numerical analysis by Triantafyllidis [131] also indicates that formation of a vertex on the yield surface can cause surface instability to occur earlier in compression than in tension, which is in agreement with what is observed here. An interesting observation mentioned before was the increased planarity of slip bands in compression. Perhaps this feature contributes to nonnormality of flow. This can also possibly cause earlier instability
by promoting the formation of a vertex at the yield surface.

Let us consider the applicability of this model to the quenched and tempered 1045 and 1090 steels. The theoretical and experimental strains for instability in these steels are shown in Table 6 and Table 7. The theoretical strains were calculated as before from the plane strain tension curves of these steels (Figures 23 and 24). Since the 1045A and 1090A fractured at very low strains in plain strain tension, it was not possible to obtain the $E_{tp}/E_{sp}$ from their curves. The theoretical strain for the 1045B steel is 0.27 and the experimental observation is 0.115 indicating some disagreement. For the 1090B steel the theoretical and experimental values are 0.295 and 0.03 indicating a large disagreement. This high disagreement in the 1090 steel can be explained in the following way. There was extensive microcracking which had developed very early in the deformation of the 1090B steel. These microcracks could have started off the instability at the observed low strains. Indeed, the presence of such inhomogeneities can cause flow localization to occur much earlier than that predicted (in the absence of inhomogeneities) using the vertex model as has been noted by Hutchinson [19] and Tvergaard [132]. Nevertheless, the theoretical strains obtained in this manner do predict that instability occurs
earlier in the high strength quenched and tempered steels as compared to the low strength spheroidized steels due to earlier formation of the vertex during yielding in the former material. Consideration of Table 7 shows that the embrittling heat treatment causes the instability strain to decrease from 0.115 in 1045B to 0.026 in 1045A steel. This effect is partly due to the higher yield strength and hence higher $E_s$ and partly due to increased microcracking as a result of embrittlement. In 1090 steel the effect of embrittlement is not that prominent due to the inherently high tendency for microcracking in this steel. This microcracking brings about early instability at a strain of about 0.03 in these steels as well as in the 1045A steel.
### Table 6

**Experimental and theoretical instability strains in sph 1090**

<table>
<thead>
<tr>
<th>Surface Instability Strain</th>
<th>Spheroidized 1090</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>U-notch in Tension</td>
</tr>
<tr>
<td>Experimental</td>
<td>0.264</td>
</tr>
<tr>
<td>Theoretical</td>
<td>0.45</td>
</tr>
</tbody>
</table>
Table 7

Instability strains in quenched and tempered 1045 and 1090

<table>
<thead>
<tr>
<th>Surface Instability Strain</th>
<th>Quenched and Tempered 1045</th>
<th>Quenched and Tempered 1090</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1045 A</td>
<td>1045 B</td>
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<tr>
<td>Experimental</td>
<td>0.026</td>
<td>0.115</td>
</tr>
<tr>
<td>Theoretical</td>
<td>0.27</td>
<td></td>
</tr>
</tbody>
</table>
Figure 22: Plane strain tension curve of spheroidized 1090 steel
Figure 23: Plane strain compression curve of spheroidized 1090 steel
Figure 24: Plane strain tension curve of quenched and tempered 1045 steel
Figure 25: Plane strain tension curve of quenched and tempered 1090 steel
4.1.2 Fracture
The spheroidized 1090 failed, by extensive shear at the notch surface brought about by surface instability and subsequent shear underneath the notch root at the bottom of the surface waves. This shearing action caused failure by void growth and coalescence along the characteristic slip traces, as was also concluded by Onyewuenyi et al [130]. The 1045A and B specimens also showed a dimpled shear region near the notch root which is indicative of the shearing action at that site. Griffis et al [10] also observed mode II cracking by shearing at the notch root in 4340 steels. But the extent of shear in the 1045 steel is much less than that in the spheroidized 1090 steel. This is attributable to more microcracking in the quenched and tempered steels accompanying the growth of the mode II region. The subsequent intergranular cracking in the 1045 steels is due to segregated impurities at the austenite grain boundaries. Thus the fracture strain is reduced from 0.31 to 0.177 in this steel due to the embrittling heat treatment. The 1090A and B steels show a mode II region near the notch root which is mostly quasicleavage in nature with some shear, indicative of the extensive microcracking during the mode II cracking. This is followed by intergranular fracture and dimpled rupture as in the 1045 steels. The 1090 steels, being inherently susceptible to
microcracking, did not show much of an effect of the segregated impurities on the fracture strain.

4.2 PRECHARGED SPECIMENS

4.2.1 Instability
In the precharged spheroidized 1090 specimens in tension, surface instability starts at a strain of 0.135 as compared to 0.264 in uncharged specimens. A similar result was obtained by Onyewuenyi et al [109] who observed that the strain for surface instability was reduced from 0.24 to 0.11 when the specimens were precharged with hydrogen. In compression, surface instability in precharged spheroidized 1090 steel starts at 0.125 as compared to 0.15 in uncharged specimens. Since instability occurs at a strain of about 0.13 in tension or in compression, microvoids either preexisting or produced during the deformation have no perceptible effect on the onset of instability in hydrogenated specimens. Hydrogen is postulated to increase resistance to cross slip [79]. Hydrogen is also postulated to decrease the Peierl's stress in iron and so hydrogen trapped at deep traps like prior austenite boundaries or carbide interfaces can enhance dislocation emission at the surface of the notch. This effect coupled with the difficulty for cross slip can promote slip planarity and possi-
bly enhance the formation of the yield vertex and promote surface instability.

With the notch in tension, hydrogen reduces the strain for surface microcracking from 0.672 to 0.31, whereas in compression the corresponding strain is reduced from 0.405 to 0.375. This drastic effect in tension and the lack of it in compression can be explained in the following way. In the tensile mode once instability occurs on the surface, the incompatibility stresses at the carbide interfaces intersecting these instability bands can become quite high. Hydrogen is also postulated to decrease the cohesive strength at carbide matrix interfaces and grain boundaries [107]. Hydrogen can also be transported by dislocations and dumped at these carbide interfaces and boundaries thus increasing the hydrogen supersaturation at these sites [55], [56]. All these effects could enhance carbide breaking and carbide decohesion or grain boundary decohesion at the notch surface. These voids that are formed can promote the growth of the instability. Indeed, Yamamoto [24] has observed that when voids are present in the instability band or outside, they promote the growth of the instability. Similar predictions have been made by Hutchinson [20] and Tvergaard [39]. These voids and the localized deformation have an autocatalytic effect in promoting each other hereonwards. In a compressive stress field, the stress
tends to close up the voids, and this stifles the growth of the instability. The effect of hydrogen in opening up voids is negated. So the instability grows at about the same rate as it does in the uncharged specimen, with a small enhancement over the uncharged case.

Thus the overall effect of hydrogen seems to be twofold. The first effect is an enhancement of the surface instability and the second effect is to open up voids which helps promote the growth of this instability deeper into the notch root into a bulk shear instability. As a result, in tension the surface cracks grow into the bulk along the characteristic slip traces accompanied by a complex mixture of quasicleavage and ductile rupture associated with the growth of the shear instability into the bulk. In compression, the shearing does lead to some void formation and cracking at and underneath the notch root and this is more so in hydrogenated specimens where the decohesive effect assists the incompatibility stresses in promoting void formation in compression.

In the quenched and tempered steels at charging currents of 15A/m², cracking seems to occur first not at the surface but underneath the notch root and at lower loads and strains than in the uncharged specimens. This cracking tendency is enhanced by the embrittling heat treatment as can be found by comparing the strains for crack initiation
at the surface in 1045A and 1045B steels. When the yield strength is high and there is an increased segregation of impurities at the grain boundaries, the effect of hydrogen is mainly to promote intergranular cracking underneath the notch root with very little effect of surface instabilities, as is observed in 1045A steels. When the hydrogen charging current is increased in this steel, it lowers the load and strain for crack initiation and fracture. Most of these steels (1045A) did not show any mode II fracture near the notch root at either current density. In a couple of specimens which showed mode II cracks, the depth of the mode II region was 0.5 mm at 15A/m$^2$ and 0.16 mm at at 80A/m$^2$ and the corresponding plastic zone depths were 0.54 mm and 0.18 mm respectively. The plastic zone depths were calculated with the aid of data on 4340 steel from the work of Griffis et al [10]. As is evident, mode I cracking began in this steel at the end of the plastic zone, where the triaxial stress is the greatest. So in this steel plastic instability does not play much of a role in promoting early crack initiation. This result would then be consistent with Troiano's theory.

But when the yield strength and segregation at the grain boundary are lower, the mode II region is much smaller than the plastic zone size. Thus in the 1045B steel the mode II regions were 0.96 mm deep at 15A/m$^2$ and 0.26 mm deep at
80A/m² and the corresponding plastic zone sizes were calculated to be 3.3 mm and 1 mm. So here mode I cracking occurs well within the plastic zone and not at the site of maximum triaxial stress, suggesting that plastic instability plays a more significant role in promoting crack initiation in this steel. This is also supported by the observation of extensive dimpled rupture in the fractographs of these 1045B steels as opposed to predominantly intergranular fracture in 1045A steels. Thus when the yield strength and segregated impurities are lower, plastic instability plays a significant role in hydrogen induced fracture. Another interesting observation in 1045B steel was that the higher charging current did not significantly decrease the loads and strains for fracture. This suggests a more prominent but indirect role of hydrogen in promoting plastic instability which in turn promotes fracture, in addition to the direct role of hydrogen in opening up voids and cracks in this steel. Similar conclusions were reached by Lee et al [116] from observations on 4340 steel. These results also agree with those of McMahon et al [118] who observed hydrogen induced plasticity at crack tips in low Mn and Si steels. But as Mn and Si were increased in the steel, the amount of intergranular cracking, in the presence of hydrogen, also increased. Similar results were also obtained in 4340 and Ni-Cr steels with varying impurity contents and hydrogen concentration by McMahon et al [117], [119], [120].
The low strains and loads for crack initiation and fracture in the 1090A steel indicate that this steel is even more susceptible to hydrogen induced cracking than the 1045A steel. Here it is apparent that at higher yield strengths the effect of segregation at the grain boundary causes a more severe degradation in the presence of hydrogen as is evidenced by the high amount of intergranular fracture observed. Higher charging currents do not cause much of an effect over what is observed at lower charging currents. It seems likely that in this case the critical combination of hydrogen concentration and stress under the notch root is easily attained at low charging currents, so that increasing the hydrogen concentration does not cause too much further reduction in fracture strains. Moreover a higher yield stress has been shown to result in a higher stress concentration at the head of a dislocation pile up at a carbide particle at the grain boundary [117]. This coupled with the weakening of the grain boundaries and carbide interfaces due to hydrogen absorption (which is enhanced by the segregated impurities) causes easy fracture in this steel. The effect of instability is not all that apparent in hydrogen induced fracture of this steel.

In 1090B steel, the strain at fracture is about the same at both low and high charging currents, but the load at fracture is somewhat higher. There is a small mode II
region seen in the fracture surface in these steels. There is also more dimpled rupture in the mode I fracture region. Since the data to calculate the plastic zone size in this steel is not available, it is not possible to compare the depth of the mode II region with size of the plastic zone at present. But the results do follow the general trend of increased plasticity, during hydrogen induced fracture, with decrease in segregation and yield strength.

In general, the quenched and tempered 1090 steels seen to be more susceptible to hydrogen induced cracking than the 1045 steels. The higher yield strength is one factor. But more significantly, the volume fraction of carbides in the 1090 steel is much higher than that in the 1045 steel. This means that there are more trapping sites available to trap hydrogen in the 1090 steel. This increase in trapped hydrogen can cause increased void, and crack initiation and earlier instability in the 1090A and B steels as compared to the 1045 steels.

The overall picture that emerges out of this discussion is that, in low strength steels the effect of hydrogen is to enhance the onset of plastic instability and promote its growth by aiding void coalescence and growth. When the yield strength is increased the effect of segregation at grain boundaries becomes important. With a high amount of segregation the steel cracks readily along prior austenite
boundaries and at carbide interfaces in the presence of hydrogen. When segregation is low, there is more of a role of plastic instability in promoting fracture in the presence of hydrogen. Extending this argument a little further, it can be speculated that when segregants are reduced to very low concentrations in high strength steel, the steel will probably fail due to an enhancement of plastic instability. A higher volume fraction of carbide will increase susceptibility to hydrogen enhanced instability and cracking.

So control of segregated impurities in steel is definitely very important. But even after these are removed there is a more inherent effect of hydrogen on plastic instability which becomes quite significant in causing premature fracture in high and low strength steels. So this factor should also be considered during design of alloys for service in hydrogen containing environments.
1. In uncharged spheroidized 1090 steel, surface instability occurs earlier in compression than in tension. This is in good agreement with the predictions based on yield surface vertex models of plasticity. The observed strains for instability are in fair agreement with the theoretical predictions based on bifurcation analysis invoking the above yield vertex concept.

2. Quenched and tempered 1045 steels are more susceptible to instability than the spheroidized 1090 steel and the observed strains for instability are in fair agreement with that predicted by the vertex model.

3. Quenched and tempered 1090 steels are the most susceptible to instability and fracture. The observed strains for instability are much lower than that predicted by the vertex model because of extensive microcracking at low strains due to the high yield strength and segregation effects.

4. Hydrogen enhances the onset of plastic instability in spheroidized 1090 steel. This effect is more drastic
in tension, because of the accompanying tendency for void formation which promotes the instability. Hydrogen also enhances the tendency for void formation and thus reduces the strain for fracture.

5. In quenched and tempered steels the effect of hydrogen in promoting cracking at grain boundaries and carbide interfaces is increased by a higher yield strength and segregation. As the strength level and the segregation in the steel is decreased, hydrogen effects in enhancing plastic instability become more significant. A higher carbide volume fraction makes the steel more susceptible to hydrogen enhanced instability and cracking due to the higher amount of trapping sites in the steel. The effect of a higher hydrogen charging current is felt more if the amount of segregation at grain boundaries and interfaces is higher.
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