A "FRACTURE LIMIT DIAGRAM" FOR DETERMINING HYDROGEN EMBRITTLEMENT OF SHEET UNDER MULTIAXIAL LOADING CONDITIONS

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Abstract

The application of punch-stretch testing as a means of determining hydrogen embrittlement of sheet under multiaxial loading conditions is described. The results may be expressed in the form of a fracture limit diagram which identifies at fracture the principal strain components in the plane of the sheet for any multiaxial deformation path associated with a thinning of the sheet. As an illustration, a fracture limit diagram is reported for internally charged, commercially pure titanium sheet. The diagram clearly shows that the titanium is susceptible to hydrogen embrittlement under plane strain and biaxial loading paths even though no loss of ductility occurs in simple tension.
Introduction

Hydrogen embrittlement (HE) has been studied extensively under conditions in which many environmental and metallurgical parameters have been controlled. However, nearly all of the studies to date rely on imposing tensile states of stress which are either uniaxial (typically tension or bending) or, on a local scale, strongly triaxial as a result of the presence of a notch or crack. Studies of HE under multiaxial stress states other than notched or pre-cracked plates have been primarily confined to disc pressure tests in which the pressure required to burst clamped metal discs is measured. However, implicit in all theories of fracture is a fracture criterion which is typically based on a critical stress, strain, or strain energy density. Although burst tests have been very useful in indicating sensitivity of HE to biaxial loading, such studies report burst pressures and not a critical stress (or strain) for fracture. Thus, our present understanding of the influence of stress or strain state on HE is limited because it must rely primarily on a comparison of two relatively extreme cases: simple tension vs. pre-cracked or notched plates.

The purpose of this communication is to describe an alternate method of determining the HE of sheet material subjected to multiaxial deformation. Based on the application of punch-stretch testing to HE, the method involves the stretching of gridded sheet over a usually hemispherical punch. The test procedure allows one to measure, sequentially or at fracture, the principal strain components in the plane of the sheet for any multiaxial deformation path associated with thinning of the sheet. Applied to HE as manifested by losses of ductility, the resulting data may be expressed in the form of a "fracture limit diagram" which identifies a strain criterion for fracture over the complete range of loading paths from uniaxial tension to balanced
biaxial tension. In two sections, this communication describes the basic aspects of the punch-stretch test method as applied to HE, and, as an illustration of the technique, preliminary results on the HE of Ti sheet under multiaxial loading conditions are reported.

**The Test Method**

Stretching is a very common mode of multiaxial deformation in sheet metal forming. As a means of assessing the stretchability of a metal, Hecker has developed a punch-stretch test in which a clamped sheet is stretched to failure over a hardened steel punch. Figure 1 schematically shows such a test. The punch itself is usually hemispherical but may be flat. The sheet is clamped securely between the die plates, usually with a V-shaped groove and matching draw beads machined into the plates to prevent drawing of the specimen. Care must be taken in HE studies so that the plane strain deformation caused by the sheet bending over the draw bead does not cause premature fracture of the specimen.

In order to determine the state of strain in the sheet either during deformation or after fracture, the specimens themselves are gridded with contacting circles or squares, which may be etched or photographically printed using a photo-sensitive resist method. Grids of this type can be applied with precision on a scale of \( \approx 0.5 \text{mm} \) and do not affect the deformation or fracture behavior of the specimen. For HE studies, care must be taken to protect the grids during testing in any environment which might degrade them or the metal substrate. The use of grids in the form of circles is especially well suited for strain measurements; from a deformed circle (ellipse), the major \((\varepsilon_1)\) and minor \((\varepsilon_2)\) principal strains in the plane of the sheet are readily determined. In the case of fracture, the magnitude of \(\varepsilon_1\) across the fractured surface can be determined either "directly" from fractured grids or "indirectly" by measuring the width strain \(\varepsilon_2\) (from the deformed grids), the thickness strain \(\varepsilon_3\), and
Fig. 1. A schematic diagram of the punch-stretch apparatus.
assuming constant volume in which case: \( \varepsilon_1 = -(\varepsilon_2 + \varepsilon_3) \). Measuring \( \varepsilon_1 \) directly from the fractured grids always introduces an error caused by any strain gradient which may exist over the scale of the grids. However, in many cases such measurements can be made with greater accuracy and are more reproducible than those based on the thickness strain which must rely on often irregular fracture surface profiles. Given a sufficiently small grid (<1 mm), experiments indicate that the two methods of obtaining \( \varepsilon_1 \) provide data showing identical trends.

Fracture of sheet specimen over the entire range of strain ratios which permit thinning can readily be achieved by controlling the degree of lubrication between the punch and the specimen or by testing strip specimens of reduced width. For example, testing a full width specimen which is well lubricated with several teflon sheets or polyurethane rubber results in loading paths near that of balanced biaxial tension \( (\varepsilon_1 = \varepsilon_2) \), such as in a hydraulic bulge test. On the other hand, testing specimens of reduced width (i.e., the specimen width is less than the punch diameter) results in a strain path between pure tension and plane strain. A series of tests of sheet specimens of various widths and under a range of lubrication conditions will determine all possible combinations of fracture strain components which are associated with thinning of the sheet.

The fracture limit diagram is simply a type of fracture map which, in terms of \( \varepsilon_1 \) and \( \varepsilon_2 \), identifies the strain components in the plane of the sheet at failure. All strain paths which cause sheet thinning are reported. This ranges from simple tension (in which case \( \varepsilon_1 = -\frac{1}{2} \varepsilon_2 \) for a plastically isotropic material), through plane strain \( (\varepsilon_2 = 0) \), and to balanced biaxial tension \( (\varepsilon_1 = \varepsilon_2) \). A fracture limit diagram for HE will thus normally consist of 1 or more data curves, one of which represents "hydrogen-free" behavior while the other(s) report failure strain in terms of \( \varepsilon_1 \) and \( \varepsilon_2 \) for the embrittled material.

In applying punch-stretch testing to HE studies, the reader should recognize certain advantages as well as disadvantages. The tests are particularly well
suited for those HE phenomena which are manifested by a loss of ductility. The loss of ductility may be a result of a decrease in the strain for the onset of localized necking (in which case local necking strains, not fracture strains, should be reported) or, more likely in the case of HE, it will reflect the intervention of a fracture prior to localized necking. In either case the state of strain at failure as well as strain distribution can be readily measured over a range of strain paths ranging from uniaxial to balanced biaxial. The test itself is most easily performed in air; as such, it is most conducive for examining specimens containing internal hydrogen. However, the die and the specimen may be used as a container for either a gaseous or aqueous hydrogen-producing environment so that an external hydrogen study may also be performed. Research of this nature is currently in progress in our department. It should be noted that, although the strain components are experimentally determined, the stress components cannot be measured because of an unknown degree of friction between the punch and the sheet. One can, however, calculate approximate values of the stresses using plasticity theory and knowing the strain components.

An Example of a Fracture Limit Diagram for Determining HE

Given the unique nature of applying punch stretching to HE studies, we shall describe an example of a fracture limit diagram which illustrates a case in which HE is sensitive to loading path. Commercially pure (CP) Ti sheet (∼0.8mm thick, 15μm grain size, and 1460 wt ppm oxygen) has been tested in an annealed condition (60 wt ppm H) and after thermal charging to 980 wt ppm H, in which case a considerable amount of titanium hydride is present. Figure 2 is a fracture limit diagram which reports the limiting fracture strains as measured directly from a 1mm square gridded element of material which includes the fracture surface. Similar data curves, but with each displaced to higher strain levels by a factor of 1.1–1.2, are obtained if thickness strains are
Fig. 2. A fracture limit diagram illustrating the influence of loading path on the hydrogen embrittlement of C.P. Ti sheet. The principal strains $\varepsilon_1$ and $\varepsilon_2$ are measured directly from fractured grid elements.
used to calculate indirectly a true fracture strain across the fracture surface.

Previous studies have shown CP Ti to be relatively immune to HE under slow strain rate, simple tension test conditions.$^{10,12}$ This is confirmed in the fracture limit diagram shown in Fig. 2 for the conditions of $\varepsilon_1$ and $\varepsilon_2$ corresponding to uniaxial tension. In contrast, Fig. 2 also indicates that a substantial loss of ductility occurs if hydrogen-charged Ti sheet is deformed under balanced biaxial tension. That HE of CP Ti can be induced by multiaxial loading is easily seen by a visual examination of the test specimens. For example, Fig. 3 shows that uniaxial tension specimens exhibit no significant loss of ductility as viewed either along the entire gauge section or measured near the fracture surface.* HE induced by biaxial straining is also clearly evident in Fig. 3. Under biaxial tension, loss of ductility, in terms of the equivalent strain to fracture $\varepsilon^*_f$, becomes substantial: $\varepsilon^*_f = 51\%$ for the Ti containing 60 ppm H but only 20% at 980 ppm H. This behavior is quite consistent with the increased notch sensitivity of CP Ti bar stock charged with hydrogen;$^{10,11}$ however, it is difficult to separate strain rate effects from stress state effects in such tests.

Figure 3 also shows a significant loss of ductility occurs in the hydrogen-charged CP Ti sheet under plane strain conditions ($\varepsilon_2 = 0$): $\varepsilon^*_f = 25\%$ at 60 ppm H but only 13% at 980 ppm H. Thus sheet metal forming operations which typically involved near-plane strain deformation, such as bending, will be deleteriously affected by this HE effect. In fact, problems of this sort have arisen and have been diagnosed in terms of the loss of ductility of CP Ti due to HE under multiaxial loading conditions.$^{14}$ Although the stress state is much different, crack-tip plasticity also depends on plane strain deformation in most cases.

*The equivalent strain at fracture $\varepsilon^*_f$ is calculated using Hill's original theory$^{13}$ given that the ratio of width strain to thickness strain in a tension test is 2.2 in the transverse direction for both materials, while in the rolling direction it is 5.5 for the sheet containing 60 ppm H and 4.6 at 980 ppm H. The magnitude of $\varepsilon^*_f$ in Fig. 3 is based on a 1 mm element of material which includes the fracture surface and is measured via fracture grids.
Fig. 3. An illustration of the influence of hydrogen on uniaxial and biaxial tensile behavior of commercially pure (C.P.) titanium sheet containing 60 and 980 ppm hydrogen. The effective strains at fracture $\bar{\varepsilon}_f$ are calculated from anisotropic plasticity theory, taking into account a moderate degree of plastic anisotropy in this material (see text.)
Figure 2 indicates that, even under the reduced plastic constraint of sheet deformation, the loss of ductility in plane strain of the hydrogen-charged CP Ti is sufficient to indicate a loss of fracture toughness due to HE of CP Ti in plate form.

The mechanism responsible for the HE of CP Ti sheet under biaxial tension conditions is at present not understood. Fractography indicates that a transgranular, ductile fracture process occurs. Given the presence of about 980 ppm H in the hydrogen-charged material, it is reasonable to speculate that an acceleration of hydride fracture under multiaxial tension causes the loss of ductility by promoting void formation. Whether hydride fracture does in fact occur at smaller equivalent strains under biaxial loading conditions is the subject of current studies.

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Hydrogen Embrittlement, Fracture Limit Diagram, Multiaxial Stress States, Commercially Pure Titanium

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