HYDROGEN EMBRITTLEMENT OF STAINLESS STEELS

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ABSTRACT

Stainless steels are specified for hydrogen service when enhanced compatibility, safety, and reliability are required. Prior to consideration of the issues surrounding containment applications, we will review the phenomenology of hydrogen effects. The mechanical response of stainless steels to hydrogen exposure varies widely as a consequence of composition, thermo-mechanical preparation, joining, and testing methods. Composition dictates phase stability and properties such as the stacking fault energy, influencing the tendency towards co-planar slip and deformation twinning. Contrary to the usual trend, stainless steels offer improved compatibility with increasing strength when the strength increment is provided through retention of warm-work, introduced through forging, followed by rapid cooling. Fusion welding processes in stainless steels introduce a readily hydrogen-embrittled ferritic (delta) phase, required for freedom from weld cracking. This phase may significantly compromise cracking resistance in hydrogen, and its' effects must be quantified and understood.

The design process for a hydrogen containment application is multifaceted, and at the completion of the process the designer should be able to certify fitness-for-purpose. The source and pressure of the hydrogen, all loads (applied and residual stresses), loading modes, conditions for plastic collapse, and possible or latent defects must be considered in order to define stresses and stress intensities. Experimental databases, developed using appropriate testing methods and applicable to the relevant conditions, both service and metallurgical, are necessary, and should include cracking sensitivity (K_{TH} in hydrogen). Performance margins may be assessed using a two-parameter Failure Assessment Diagram (FAD). In this approach, a failure envelope is defined using normalized loads and normalized stress intensities, defining linear elastic, transition, and plastic collapse regimes as load increases. In this context, the R6 FAD approach to hydrogen embrittlement of austenitic stainless steels offers a means of specifying fitness-for-purpose. R6 requires validation of the approach for the case of hydrogen embrittlement. The presence of deformation mode changes, fracture mode changes, and the substitution of the cracking threshold (K_{TH}) for the linear elastic fracture toughness (K_{Ic}) also require validation of the approach. The choice of margins (factors of safety against cracking and plastic collapse) is then made, defining the "safe" operating regime. We will discuss those issues, and present the results of both experimental and computational development of the failure assessment curve for specific examples of stainless steels tested in a hydrogen embrittlement environment.

1. INTRODUCTION

Hydrogen embrittlement of structural steels is known to cause early failure at lower loads and shorter times than without exposure to hydrogen, effects that were first observed almost 130 years ago. The potential result of this phenomenon is catastrophic failure with loss of property and life. Consequently, much effort and many publications (greater than 3000) have been devoted to characterizing and defining the driving forces, the metallurgical factors affecting embrittlement, and to understanding the mechanisms of embrittlement. The source of hydrogen (determining fugacity and location), the temperature and temperature history, and the duration of hydrogen exposure define the potential severity of hydrogen embrittlement. Metallurgical and mechanical factors then determine the response of the structure to the environment. (We will not consider hydrogen attack, a characteristic of high temperature service conditions.) Austenitic stainless steels are commonly more compatible with hydrogen-bearing environments than ferritic alloys, and the austenitic stainless steels are often specified for hydrogen service when their enhanced compatibility, safety, and reliability override their expense. In this paper we will focus on the austenitic stainless steels, including nitrogen-strengthened variants, dealing first with the phenomenology of hydrogen embrittlement, and correlations with metallurgical factors. We will review recent contributions to the understanding of mechanisms of hydrogen embrittlement in austenite. Finally we will discuss progress in the application of one of the most modern and important methods for assessing performance margins and fitness-for-purpose, as applied to stainless steels. That method is based upon failure assessment diagrams.

2. HYDROGEN EMBRITTLEMENT PHENOMENOLOGY

The phenomenology of hydrogen embrittlement in austenitic stainless steels has been extensively described, and we will review the essentials. Mechanical properties studies have used tensile testing, fracture mechanics measurements of cracking threshold (both rising load and sustained load), fatigue, and impact testing. The tensile yield strength of an austenitic stainless steel typically increases only slightly or may decrease slightly when specimens are pre-charged with hydrogen [Louthan et al., 1, West and Holbrook 2, Holbrook and West 3]. Ultimate tensile strength may not change, or decrease slightly, [Louthan et. al, West and Holbrook]. Typical embrittlement effects are a reduction in ductility, described as the % reduction of area loss (%RAL) at fracture. In smooth bar tensile tests of base metals at room temperature, steels such as AISI 310, 316, 309S, Nitronics®40(21Cr-6Ni-9Mn) and Nitronics®50 (22Cr-13Ni-5Mn) show minimal losses (10-25%), while 304L shows a significant loss, that is about 60% RAL when thermally charged and tested in hydrogen [West and Holbrook]. When comparing notched tensile specimens, 304L exhibits a 40% RAL and 21-6-9 exhibits a 36% loss[West and Holbrook]. Test techniques must be considered when comparing results. For example, specimens internally charged with hydrogen and tested in hydrogen typically exhibit more significant degradation than exhibited by environmental hydrogen exposure alone.

Embrittlement effects are typically confined to near-ambient temperatures, maximizing near room temperature and diminishing at higher or lower temperatures. A notable anomaly is Nitronics ®40 which exhibits a ductility minimum at about 250K with improved properties above and below that temperature[Holbrook and West, and Caskey 4]. This phenomenon may be related to deformation twinning. By comparison, 304L exhibits a continuous decrease in ductility below 300K down to about 210K. Below 210K, hydrogen effects begin to disappear [Holbrook and West]. Maximum embrittlement is also normally observed at slow tensile deformation rates of the order 10^{-3}- 10^{-5} s^{-1}, diminishing at higher rates. There are exceptions to this statement: 304L pre-charged with hydrogen by low temperature thermal means may show a 50% loss in impact energy absorption in testing at 77K, when compared to room temperature tests in which impact energy is little affected by hydrogen [5. Martensite formation was not involved in the loss of energy absorption capability; rather, microvoid coalescence (MVC) initiated at very fine inclusions caused the void formation and subsequent void-sheet like failure [Hyzak et al.].

Fracture mode changes are observed as a consequence of hydrogen embrittlement, although tensile fracture mode changes are not a sufficient test for hydrogen embrittlement. Fracture normally occurs in air by MVC in which inclusions separate from the matrix and the resulting voids grow and coalesce. In hydrogen, fracture may occur through either accelerated microvoid growth or coalescence, in which the density of fracture surface dimples is decreased, or by accelerated microvoid nucleation, in which the density of fracture surface dimples is increased. Severe ductility loss is usually correlated with extreme changes in microvoid size, either smaller or larger, indicating either enhanced void nucleation or growth[6] An intergranular mode may be also observed, such as in highly sensitized 304 which undergoes phase transformation to martensite at grain boundaries (caused by chromium depletion at grain boundaries and an elevation of the deformation-induced martensite transformation temperature M_D), causing very high rates of cracking[7]. Transgranular slip band fracture in Nitronics®40 has been observed at low (0.27 w %) nitrogen concentration, giving way to twin plane parting and intergranular secondary cracks with increasing nitrogen concentration. At high (0.47 w %) nitrogen concentration, the strength increase is accompanied by an increasing amount of intergranular fracture, although the %RAL does not decrease [Holbrook and West] Under conditions of cathodic charging, superficial grain boundary failure has been observed in laboratory heats of annealed 304L having very low carbon[8]. Often, mixed behaviors are observed [Holbrook and West] In addition, twin boundary parting of annealing twins has been observed in annealed 304L, Nitronics®40 and other steels [Holbrook and West, West and Holbrook, Caskey]. In welded materials containing ferrite, separation of the austenite/ferrite boundary is observed in tensile testing[9] Sensitization may greatly enhance the extent of hydrogen embrittlement; 304 exhibits intergranular fracture [Briant] while

304L is not readily sensitized. Nitronics®40 [West and Louthan 10] exhibits only a 25% increase in %RAL when sensitized, thermally charged and tested in high pressure hydrogen.

Resistance to sustained load cracking (Slow Crack Growth or SCG threshold) differs significantly among the steels. 304L did not crack at a stress intensity of 120 MPa√m and a hydrogen pressure of 207 MPa and Nitronic®40 cracked at 100 MPa√m [Perra,11], while another study reported slow crack growth in pre-charged Single Edge Notch specimens of 304 [Sing and Altstetter 12]. Stable steels such as 310 have been reported to crack when thin specimens are tested in hydrogen [13]. Perra also reported an "upper bound" inverse linear relationship between yield strength and SCG threshold above 700 MPa in stainless steels having "clean" grain boundaries. Steels exhibiting grain boundary particulate or second phases exhibit lower SCG threshold values than the upper bound. Fusion welded microstructures exhibit variable behaviors depending upon the base material and the quantity of ferrite in the weld. The value of K_{Ic} (from J-integral testing in which $J = \sqrt{KE})/(1-v^2))$ increases compared to a forged base metal in welds tested in air, probably due to decreased strength in the welds. Welds then exhibit substantial losses equal or greater than that of the base metal K_{TH} value when welds with ferrite content above about 8% is exposed to hydrogen [Morgan, 14].

Briant [7] cites studies of stainless steels with yield strengths varying from 200 to 600 MPa, demonstrating that the %RAL when tensile tested in 69 MPa hydrogen, increases with yield strength. Nevertheless, it is clear that microstructure is "the more fundamental" contributor to properties in hydrogen [Bernstein,15] and must be considered in the austenitic steels. Some examples follow.

Compositional effects on the mechanical properties in hydrogen are linked to the deformation modes. Typically, the more stable the austenite phase, the better the mechanical properties in hydrogen. Stacking fault energy (SFE) determines the slip mode, which affects stress concentrations and therefore hydrogen embrittlement. Low SFE favors coplanar slip and deformation twinning, whereas cross slip is favored at high SFE. Nitrogen in particular exerts a potent effect on SFE and therefore slip mode. As reported for Nitronics®40 a loss of %RA occurs coincident with a reduction in SFE above 0.3 wt% nitrogen[16]. Mullner et al. [17] identify manganese and nitrogen as promoters of brittle fracture in austenitic steels. Nitronics®50 however is resistant to this change, since the SFE is high enough that coplanar slip does not develop[Caskey].

The effect of SFE and deformation modes may be compared in both hydrogen and tritium effects studies on Nitronics®40 and Nitronics®50. When tested in hydrogen, Nitronics®40 experiences a severe ductility dip at 200-250K while Nitronics®50 does not [Holbrook and West, Caskey]. Tritium, the radioactive isotope of hydrogen, differs from hydrogen in its' rapid decay to helium-3 at the rate of 5.5% per year (half life ~ 12.33 years). Tritium diffusively permeates the steels, as does hydrogen, differing only in its' slower rate by $(1/\sqrt{3})$ and its' decay to helium-3 within the metal. The accumulation of helium results in the formation of small, highly pressurized bubbles, and subsequent increases in the yield strength of the alloy [Robinson and Thomas 18, Robinson, 19]. As a result of extensive tritium exposure, Nitronics ®40 exhibits extensive deformation twinning [Robinson and Thomas], whereas Nitronics®50 is resistant to both twinning and loss of properties under the same conditions[20]. The mechanism in tritium-affected property loss appears to be helium bubble pinning of dislocations, inhibiting slip and forcing deformation twinning to occur at small strains. Intergranular fracture dominates at high helium concentration [19-21], occurring at the intersections of deformation twins with grain boundaries. Fracture is assisted by hydrogen, and by helium bubbles on the grain boundary. Removing the hydrogen isotope recovers ductility, and the fracture mode reverts to MVC, while the yield strength increment is retained. This form of hydrogen embrittlement may be described as the hydrogen embrittlement of a metal strengthened by helium bubble precipitation.

3. TESTING METHODS

The mode of hydrogen exposure, in particular the method of pre-charging to produce internal hydrogen, may influence testing results; cathodic charging is capable of introducing extraordinarily high concentrations of hydrogen at the surface simply by increasing the galvanostatically controlled current density and may be enhanced by introducing specific "recombination" poisons. For example, IN903, an iron-base superalloy, will absorb as much as one atom of hydrogen per metal atom at 100 mA/cm2, when electrolytically charged in a sodium arsenate-poisoned bath of 1N sulfuric acid [Robinson et al, 21]. Nevertheless, low temperature cathodic charging coupled with low hydrogen diffusivity ($D \sim 10^{-16}$ m^2/s or smaller at room temperature) in the stainless steels biases results towards surface effects. (This effect was observed in the previously discussed experiments on the role of martensite formation on fracture modes.) Careful control of charging and testing procedures is required to avoid superficial cracking due to surface volumetric changes following cessation of charging. Elevated temperature cathodic charging in salt solutions has been more successful in providing uniformity of hydrogen pre-charging [Singh and Altstetter]. Samples must be quenched to retain hydrogen, and low to room temperature testing may then be performed with only superficial loss of hydrogen. Elevated temperature gas phase charging, although not capable of achieving high concentrations, allows full saturation of reasonably sized specimens with the penalties of somewhat extended charging times, the requirements for high pressure safety, and the potential complications arising from the small loss of surface hydrogen during removal of specimens from the charging apparatus.

A significant applications issue is the use of accelerated testing methods to obtain minimum cracking thresholds properties. Mode I thresholds from environmental slow crack growth testing of a high strength austenitic superalloy, IN903, may be correlated with rising load fracture tests (K_{TH} and K_{IC}), when local hydrogen concentration and critical fracture modes are considered [Moody et al, 22]. These results suggest that hydrogen concentration enhancement in the fracture process zone is necessary for minimal properties, and that accelerated testing using only precharging of hydrogen may not always be conservative. This correlation has not been experimentally examined in the stainless steels.

Mode I has commonly been considered in fracture mechanics studies to be the most damaging, to the exclusion of considering other modes. This may not be true in the case of environmentally degraded cracking. Shear deformation in mixed modes I and II may introduce microstructural change (damage) such as slip and deformation twinning, reducing toughness, as reported for an austenitic steel in the absence of hydrogen [Jeon et al., 23]. Thus interaction between modes may result in a minimum in a mixed loading situation, in which the shear mode generates microstructural damage and Mode I provides the necessary crack tip opening driving force. Original research in this area will be discussed using an innovative approach applied to a common bend specimen design.

4. HYDROGEN EMBRITTLEMENT MECHANISMS

The currently accepted and competing models of hydrogen embrittlement are, HELP (<u>H</u>ydrogen <u>E</u>nhanced <u>L</u>ocal <u>P</u>lasticity), decohesion, and hydride formation. The effect of solute hydrogen on slip and twinning modes has been examined [Moody and Robinson 24]. Slip band spacings in IN903 exhibit no change with hydrogen charging. Furthermore, twin nucleation strain in hydrogen-charged Nitronics®40 does not change with hydrogen charging. The belief that hydrogen raises M_D thereby causing accelerated failure through a martensitic phase has been voiced several times. The stable steels (such as AISI 310) have generally been immune to hydrogen cracking. However, hydrogen pre-charged, very thin (2 mm) plane stress fracture specimens of nominally stable 310 and 321 have exhibited cracking [Chu et al., 25]. The 321 specimens showed alpha prime as a result of charging, while 310 exhibited formation of epsilon martensite. Dislocation spacing and strain field shielding effects (the HELP model) were modeled in 1989 [Birnbaum, 26]. Extensive evidence based upon transmission electron microscopy has now been published [Ferreira et al, 27], showing closer spacing in dislocation pileups. Modeling has shown reduced strain fields around dislocations in the presence of hydrogen, resulting in enhanced dislocation mobility, slip localization and reduced dislocation spacing [Birnbaum]. Thus HELP has now assumed a strong role in explaining accelerated fracture by hydrogen enhancement of localized deformation and fracture. The decohesion model predicts that surface energy must decrease with hydrogen adsorption, reducing the work of separation. Hydrogen induced intergranular fracture and twin plane parting have been attributed to decohesion. Birnbaum has proposed that the evidence for plastic deformation even in intergranular fracture argues against decohesion. Hydride formation is not believed to be a concern in the austenitic stainless steels and is not treated further.

The failure criterion embodied in the Rice-Knott-Ritchie (RKR) [29] model, that fracture occurs when the applied normal stress equals or exceeds the fracture stress over a critical distance, has been the most successful model of crack initiation. It is commonly assumed that hydrogen transport is required, in order to enhance local hydrogen concentrations, however, hydrogen supersaturations are unlikely to result from this mechanism[Birnbaum]. Furthermore, deformation enhanced hydrogen supersaturations have been shown unnecessary to explain many (but not all, as we observed in the case of slow crack growth) embrittlement phenomena such as those of Hyzak et al. Taha and Sofronis [30] have treated crack tip hydrogen enhancement, identifying concentration enhancements in the near-surface region of extensive plastic deformation, and a deeper region of hydrostatic dilation. Unfortunately only the extremes of low and high flow stress were considered, while most practical materials are used at intermediate strength levels. Experimental observations on nickel [Sun et al 31] in which the two zones of crack tip enhancement were observed, are awaiting confirmation.

5. APPLICATION OF FAILURE ASSESSMENT DIAGRAMS

The design process for a hydrogen containment application is multifaceted. Hydrogen concentrations, loads (applied and residual stresses), loading modes, conditions for plastic collapse, and possible or latent defects should be considered to define stresses and stress intensities. Experimental databases of material performance, developed using appropriate and relevant testing methods are required, including cracking sensitivity (K_{TH} in hydrogen). The designer should then be able to certify fitness-for-purpose, and the R6 Failure Assessment Diagram (FAD) [32] approach to hydrogen embrittlement of austenitic stainless steels offers a means of specifying fitness-for-purpose. In this approach, a failure envelope is defined using normalized loads and normalized stress intensities, defining linear elastic, transition, and plastic collapse regimes. With a validated FAD, the choice of margins (factors of safety against cracking and plastic collapse) is then made, defining the "safe" operating regime. However, the R6 revision 4 standard requires validation of the approach for the case of hydrogen embrittlement. The issues include, deformation mode changes, fracture mode changes, and the substitution of the cracking threshold (K_{TH}) for the linear elastic fracture toughness (K_{Ic}). We have observed that, in the Option 2 approach, increasing the yield strength whether by warm-working or by tritium-aging extends the linear elastic regime of the FAD, and the failure curve approaches that of the Dugdale strip yield analysis, which is appropriate for a non-hardening material. This result is consistent with the elevated yield strength and reduced hardening observed in tritium-affected stainless steels. The effect of a hydrogen-induced fracture mode change is being addressed using the Option 3 method through a fracture mechanics approach, testing an austenitic superalloy alloy known to exhibit intergranular fracture above a critical hydrogen concentration. We are extending the work to annealed and forged 304L steel, and examining the effects of mixed mode loading. We will discuss those issues, and present the results of both experimental and computational development of the failure assessment curve for specific examples of stainless steels tested in a hydrogen embrittlement environment.

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