Quarterly Progress Report

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Project Title: Task 2, Enabling Hydrogen Embrittlement Modeling of Structural Steels

Project Period: July 1, 2010 to September 30, 2010
Date of Report: April 9, 2010

Principal Investigator: Brian Somerday, 925-294-3141, bpsomer@sandia.gov
Other Key National Lab Researchers: Chris San Marchi (SNL/CA), Kevin Nibur (SNL/CA)

Sub-Contractors Funded through AOP Task: none

Industrial Partners: Secat, University of Illinois (Petros Sofronis), NIST (David McColskey)

DOE Managers: Sara Dillich, Hydrogen Production & Delivery Team Lead
Rick Balthaser, DOE Field Project Officer

Project Objective: The principal objective of this project is to provide an experimental component to the development of prognosis models for steel hydrogen gas pipelines. These models include both mechanism-based simulations of hydrogen embrittlement as well as structural integrity analyses to predict safety margins for pipelines. The aim of the experimental effort is to establish physical models of hydrogen embrittlement in steels and to generate material properties that serve as model inputs. The focus of the latter is on fracture mechanics properties such as crack propagation thresholds and fatigue crack growth relationships.

Background: Carbon-manganese steels are candidates for the structural materials in hydrogen gas pipelines, however it is well known that these steels are susceptible to hydrogen embrittlement. While hydrogen embrittlement compromises the structural integrity of steel components, decades of research and industrial experience have allowed many salient variables that affect hydrogen embrittlement of steels to be identified. As a result, there are established paths for managing hydrogen embrittlement in steels and quantifying safety margins for steel hydrogen containment structures. For example, fatigue crack growth aided by hydrogen embrittlement is a potential failure mode for steel hydrogen containment structures subjected to pressure cycling. Applying appropriate structural integrity models coupled with measurement of relevant material properties allows quantification of safety margins against fatigue crack growth in hydrogen containment structures.

Status: One of the principal efforts during FY10 Q4 was evaluating the effects of load-cycle frequency on the fatigue crack growth relationship for X52 line pipe steel in hydrogen gas. The fatigue crack growth rate (da/dN) *vs* stress-intensity factor range (ΔK) relationship is a necessary material-property input into damage-tolerant life prediction models applied to steel hydrogen pipelines. One such design life methodology for steel hydrogen pipelines was recently published in the ASME B31.12 code. The measurements of crack propagation thresholds and fatigue crack growth relationships in this task support the continued optimization of the materials testing methods in the ASME code, i.e., enhancing the efficiency of the test methods without compromising data reliability.

The X52 line pipe steel was selected for this task because of its recognized technological relevance for hydrogen pipelines. Additional material from the round robin tensile property study (FY08) was procured for testing. This X52 steel is featured for the following reasons: 1) some characterization of the material was already provided from the round robin study, 2) ample quantities of material were still available from CTC, one of the participants in the Pipeline Working Group, and 3) the X52 steel was in the form of finished pipe, which is the most relevant product form and also allows samples to be extracted from the seam weld.

The hydrogen-affected fatigue crack growth relationship (da/dN *vs* ΔK) for the structural steel is the basic element in pipeline life-prediction models. The ASME B31.12 code requires measurement of the fatigue crack growth relationship for pipeline steels at the hydrogen gas operating pressure. Initial measurements of the fatigue crack growth relationship for X52 steel were conducted in 21 MPa hydrogen gas (the upper limit specified for hydrogen pipelines in the ASME B31.12 code) at a load-cycle frequency of 1 Hz (Figure 1). This load-cycle frequency was selected to balance test effectiveness and test efficiency, since fatigue crack growth rates can be enhanced at lower test frequency but the test duration can become prohibitively protracted. Even at this relatively high load-cycle frequency, measurement of the fatigue crack growth relationship over the relevant range of ΔK could require several days. For example, the duration of the test conducted at a load ratio of 0.5 was 6 days. (Load ratio, R, is the ratio of minimum applied load to maximum applied load.)

Although the accepted trend is that increasing load-cycle frequency leads to lower (i.e., non-conservative) fatigue crack growth rates in hydrogen gas, this trend is predominantly based on fatigue crack growth rates measured for steels at relatively high ΔK, e.g., greater than 15 MPa $m^{1/2}$. The possibility of effectively measuring fatigue crack growth rates at high load-cycle frequency in the lower (and technologically relevant) range of ΔK was explored for the X52 steel in 21 MPa hydrogen gas. Fatigue crack growth rate relationships were measured at 10 Hz for two R ratios (0.1 and 0.5), and the results are compared to the fatigue crack growth relationships measured at 1 Hz in Figure 1. Although the fatigue crack growth relationships at 1 Hz and 10 Hz are not exactly coincident, there are only moderate differences in the fatigue crack growth rates over the ΔK range investigated. These preliminary results suggest that reliable fatigue crack growth relationships may be measured at high load-cycle frequency, which would allow the testing to be conducted more efficiently.

In addition to the fatigue crack growth relationship, the design life methodology in ASME B31.12 requires measurement of the threshold for rapid crack extension in hydrogen gas under quasi-static loading. Recent results from the Materials Compatibility task in the Safety, Codes and Standards project at Sandia indicate that fracture threshold measurements under rising-displacement loading may yield more conservative values compared to measurements under static-displacement loading. As such, the fracture thresholds for X52 were measured under rising-displacement loading at rates of approximately 0.0051 and 0.051 mm/min. Similar to the issue with load-cycle frequency in fatigue crack growth rate testing in hydrogen gas, the optimum loading rate that balances test efficiency and data reliability must be established for fracture threshold testing. Figure 2 suggests that the fracture thresholds for X52 steel are not sensitive to loading rate over the range explored. Although additional measurements must be conducted at higher and lower rates to conclusively establish the trend, these preliminary results suggest that reliable fracture thresholds may be measured at a relatively high rate, i.e., total testing time less than 1 hour.

Measurements of the fatigue crack growth relationship and fracture thresholds for X52 steel have only been conducted on the base metal. However, it is important to measure these properties for welds, since welds may be more vulnerable to hydrogen-assisted fracture. Specimens for measuring the fatigue crack growth relationship for the electric resistance weld (ERW) in the X52 line pipe are being prepared. This specimen preparation process has presented some challenges, since it has been difficult to reveal the ERW with chemical etching. It is important to clearly reveal the ERW so that the fatigue crack growth specimens can be precisely located on either the weld bond line or in the weld heat-affected zone (HAZ). Completion of specimen preparation and results from preliminary fatigue crack growth testing in hydrogen gas are expected during FY11 Q1.

Plans for Next Quarter and Key Issues: The focus for FY11 Q1 is measuring the fatigue crack growth relationships for X52 seam welds and girth welds in hydrogen gas. Currently, steels with girth welds have not been procured, however two possible sources have been identified.

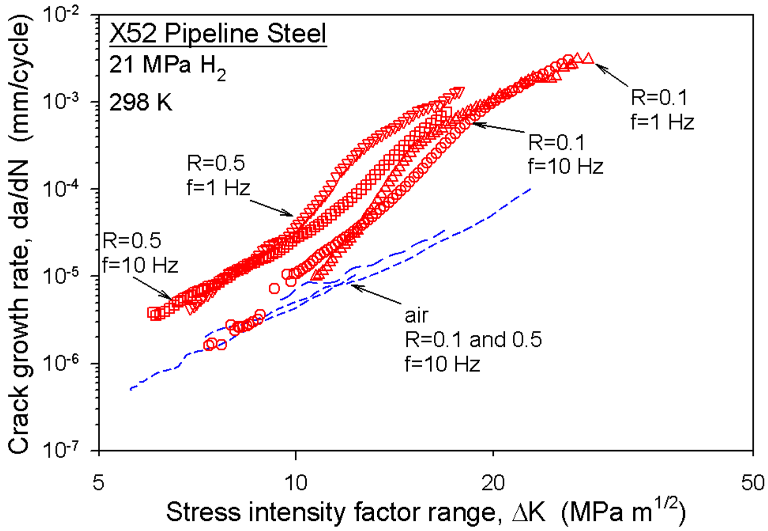

Figure 1. Fatigue crack growth rate (da/dN) *vs* stress-intensity factor range (ΔK) plots for X52 steel in hydrogen gas and air.

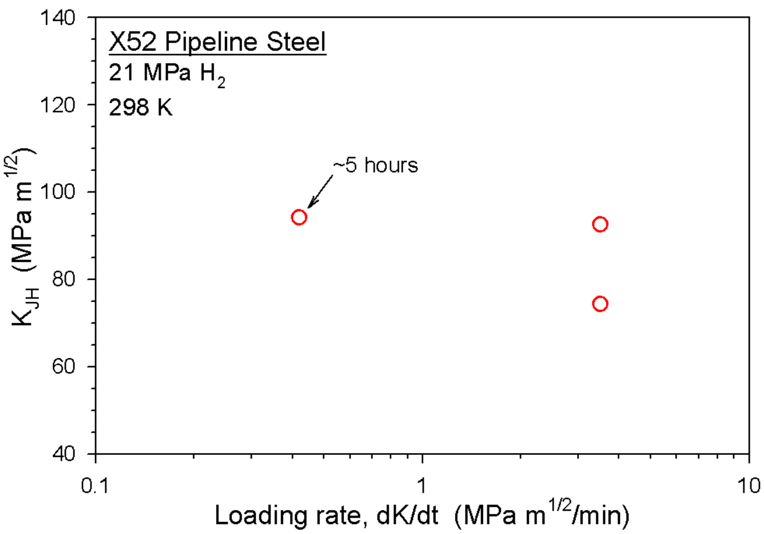

Figure 2. Fracture thresholds measured for X52 steel in 21 MPa hydrogen gas at two rising-displacement rates.

Publications / Presentations:

"Microstructure and Mechanical Property Performance of Commercial Grade API Pipeline Steels in High Pressure Gaseous Hydrogen", D. Stalheim, T. Boggess, C. San Marchi, S. Jansto, B. Somerday, G. Muralidharan, P. Sofronis, *Proceedings of IPC 2010 8th International Pipeline Conference*, Calgary, Alberta, Sept. 27-Oct. 1, 2010, IPC2010-31301.

.

Task/Milestone Schedule

Task Number	Project Milestones	Task Completion Date				Progress Notes
		Original Plan	Revised Planned	Actual	Percent Complete	
2.1.1	Measure values of K_{IH} for X52 steel base material as a function of displacement rate at constant H_2 gas pressure	06/30/10			50%	Testing completed at two rates
2.1.2	Measure the da/dN vs ΔK relationship for X52 base material as a function of cyclic load frequency at constant H_2 gas pressure	03/31/10			100%	Replicate tests recommended
2.1.2	Measure the da/dN vs ΔK relationship for heat-affected zone in X52 seam weld at constant cyclic load frequency and H_2 gas pressure	09/30/10			10%	Specimen preparation in progress
2.1.2	Develop specimen geometry and test method for measuring the da/dN vs ΔK relationship for girth welds in H_2 gas	09/30/10			0%	Need weld materials