Statement of Research

Scientific Importance

Hydrogen has long been considered an abundant and carbon-free candidate for transforming our energy infrastructure towards more renewable and alternative technologies [1]. However, ferritic steels, which are widely used in pipelines are particularly susceptible to hydrogen embrittlement [2]. Pipelines require a variety of welding processes to be assembled and repaired, which can result in complex microstructures, high residual stress, and changes in mechanical properties that affect how and where cracks initiate and propagate. Therefore, understanding how all these factors contribute to crack growth and fracture resistance of materials related to steel infrastructure is of great interest, particularly when exposed to hydrogen.

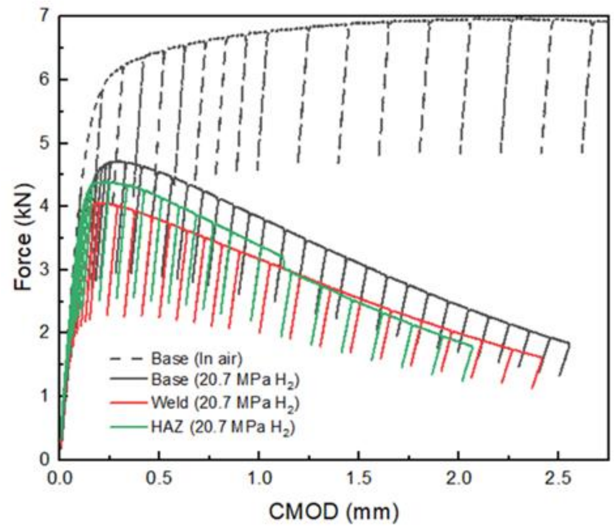

Figure 1: Force-CMOD curves collected from of X65 steel base metal, HAZ, and weld measured in 20.7 MPa hydrogen in addition to an in-air base

The fatigue and fracture group at NIST-Boulder has conducted comprehensive testing, funded by the DOT Pipeline and Hazardous Materials Safety Administration (PHMSA) [3], to identify correlations between such factors and their influence on crack growth, with more recent studies focusing on the role of residual stresses. However, establishing such correlations can be difficult. For example, there can be a disconnect between results obtained through large-scale testing of a pipeline *vs*. small-scale testing of machined and geometrically constrained specimens. To address these concerns, in March 2026 our team performed neutron diffraction measurements on fracture toughness specimens using the High Intensity Diffractometer for Residual Stress Analysis (HIDRA) to determine residual stress relief that is exhibited from steel welds as a result of sample preparation. This proposal aims to build off of our previous work by measuring the redistribution of residual stress as a result of a propagating crack in the presence of hydrogen during in-situ fracture toughness testing. This work has both scientific importance, by elucidating the impact of residual stress on the fracture resistance of pipeline steels, as well as anticipated impact on standards by providing the data necessary for updating fracture testing standards (e.g. ASTM E1820, CSA CHMC-1) to incorporate residual stresses.

Preliminary Work

NIST-Boulder has performed extensive mechanical testing and characterization as a part of the larger-scale DOT/PHMSA project [3]. Tests include Charpy impact, hardness mapping, microstructure characterization, residual stress, and fracture toughness (FT), with the latter performed in 20.7 MPa hydrogen gas. Figure 1 shows force *vs*. crack mouth opening displacement (CMOD) curves obtained from FT testing of a pipeline weld, base metal, and heat-affected zone (HAZ) in the presents of hydrogen. Additionally, an in-air test of the base metal is shown for comparison. It is apparent that hydrogen greatly decreases the ductility of the pipeline steel weld, and when analyzed in accordance to ASTM E1820 [4], results in low fracture toughness values ranging from 75 – 110 MPa*m$^{1/2}$ [5].

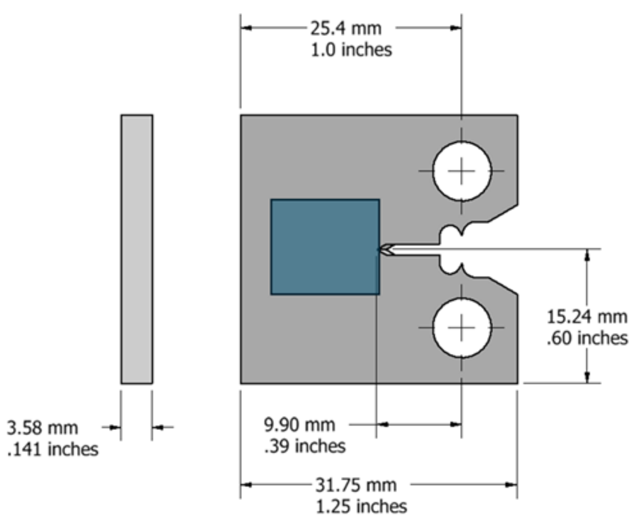

Figure 2: Compact tension C(T) engineering drawing of a thin fracture toughness specimen. Blue region indicates area for neutron diffraction scans.

We wish to expand on our previous studies of mapping the residual stress in pipeline welds (IPTS - 32131) and machined specimens (IPTS – 34181) by now performing in-situ fracture toughness testing on HIRDA under a hydrogen atmosphere. This can be achieved by integrating the 20 kN load frame designed for HIDRA, and a custom aluminum pressure chamber developed by NIST for operation at 3.45 MPa (500 psi) hydrogen, and specifically designed for X-ray and neutron studies [6]. This chamber has been used on an X-ray beamline to map strain and dislocation density in fatigue specimens of similar dimensions to the one shown in Figure 2 [7]. However, due to the limited penetration depth of X-rays in steel these experiments were constrained to thin steel specimens and were unable to probe the multi-dimensional strain components needed for calculating stress. Moreover, these measurements were limited to a small area near the crack tip whereas with neutron diffraction and large gauge volumes, it is possible to map larger areas providing a macroscopic view of the stress distribution.

Figure 3 shows detailed stress map in the hoop direction obtained from a 1 ft. x 1 ft. section of a 0.5-inch thick X65 steel weld measured on HIDRA [3,5]. This stress map is overlayed onto an optical image of the weld, which helps identify regions of high tensile stress along the fusion line. In early March 2026, our team will be measuring to what degree these stresses are relieved from the weld as a result of specimen preparation. Based on this preliminary data, we seek to determine to what extent do these stresses remain in the weld after specimen machining and pre-cracking. With this current proposal, we aim to use the stress maps obtained from machined specimens as the initial conditions for determining, **how does crack growth during a fracture toughness test affect the redistribution of residual stresses?**

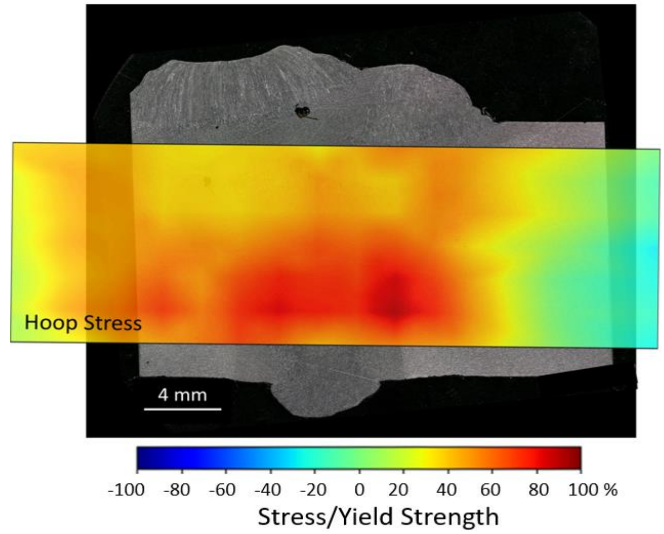

Figure 3: Map of residual stress in the hoop direction of a X65 girth weld. A slight transparency has been applied to the stress map so the optical image of the weld can be seen for clarity.

Choice of Instrument

HIDRA is optimized for resolving residual stresses in steel welds, thanks to the highly penetrating nature of the neutron. The PI has previous experience on HIDRA and has used slits to probe gauge volumes as small as of 2 x 3 x 2 mm^3. The neutron flux, overlapping gauge volumes, and length scales accessible on HIDRA make resolving regions of high/low tensile stress in weld and HAZ possible (see Figure 3). In the event beamtime cannot be allocated for HIDRA, an alternative instrument capable of measuring precise shifts in lattice strain at ORNL would be VULCAN.

Experiment Plan

We request 6 days of beamtime to perform in-situ fracture toughness testing on a total of 4 girth weld C(T) specimens on HIDRA. These specimens will be fabricated using high and low heat inputs to impart variable initial conditions of the residual stress. In-air measurements will be performed on each, with an additional 2 measurements taking place in a 3.45 MPa (500 psi) hydrogen environment. In addition, 2 comb-like specimens will be measured for determining the strain-free condition (*i.e.* d_0 sample) of the high and low heat input weld. Machined and pre-cracked specimens will possess a C(T) geometry similar to the one shown in Figure 2, but with a thicker dimension that will be suitable for testing on the 20 kN load frame available on HIRDA. The BCC Fe (211) diffraction peak will be used to measure changes in lattice strain from the radial and hoop directions of each specimen. Given the dimensions of the C(T) specimens, the PI's previous experience on HIDRA, and discussions with beam scientists, a 2 x 10 mm incident beam, and a 2 mm diffraction slit will be used with count times of 60 seconds per point. A grid of 17 x 5 points centered on the target zone (blue box in Figure 2) will be collected from the two orthogonal directions resulting in an approximate count time of 1.5 hours per sample orientation. With a minimum of two orientations and 6 measurements at various hold times in the force *vs*. CMOD curve (see figure 1), a total counting time of 18 – 24 hours is estimated for completion of one data set. Because additional time will be needed for sample change outs and purging of gases to the chamber, we estimate 6 days is sufficient to measure the 4 samples discussed.

Data Analysis and Scientific Outcomes

Lattice strains inferred from d_{211}-spacing shifts measured near the crack tip from the radial and hoop directions of the welds will be analyzed using the pyRS software suite on HIRDA to obtain residual stress maps, in addition to other peak parameters. Understanding how residual stress changes during crack growth in air, contrasted with result in the presence of hydrogen, has the potential to greatly impact interpretation of mechanical testing data related to embrittlement. Identifying correlations between residual stress, hardness, and FT will be critical in updating hydrogen pipeline codes and standards to provide U.S. industry recommendations of welding procedures and microstructures suitable for hydrogen service.

Safety Considerations

The primary safety concern would be the use of hydrogen gas. The aluminum chamber [6] is rated to a moderate pressure operation of 3.45 MPa (500 psi). The proposal team have successfully utilized this chamber at these hydrogen pressures at Argonne National Lab and NIST's Center for Neutron Research under an existing Standard Operating Procedure (SOP). A hazard review of the SOP will be conducted with close coordination with beam scientists. Additionally, operation of the 20 kN load frame is a safety concern. Strict detector limits will be applied to sensors to ensure safe operation of the load frame, and with the use of hydrogen gas, a team member will always be present as to not allow testing to go unmonitored.

References

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