CAAP Annual Report

Date of Report: *10/07/2024*

Prepared for: *U.S. DOT Pipeline and Hazardous Materials Safety Administration*

Annual Period: **From** *(09, 30, 2023)* **to** *(09, 29, 2024)*

Contract Number: *693JK32250004CAAP*

Project Title: *Development of Compatibility Assessment Model for Existing Pipelines for Handling Hydrogen-Containing Natural Gas*

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Section A: Business and Activities

(a) Contract Activities

- Contract Modifications:
  - A revised budget with narratives has been submitted for approval. This updated version addresses and eliminates the discrepancies that were present in some of the original budget lines, ensuring that the revised figures are now in full alignment with the overall project budget.
- Educational Activities:
  - Student mentoring: The Principal Investigators (PI and Co-PI) are currently supervising four graduate and one undergraduate research assistant. The students receive guidance on study techniques, time management, energy transition, and the challenges of hydrogen embrittlement related to repurposing natural gas pipelines.
  - Student internship: The PI and Co-PI assist students in exploring career options, setting goals, and building the skills and networks essential for future success in the energy field. They encourage participation in career fairs and job search activities to secure summer internships. In 2024, two students completed their internships and received full-time job offers. Additionally, the PI and Co-PI help students develop self-confidence, resilience, and other important life skills.
- Dissemination of Project Outcomes:
  - Journal Publications: Our research team is dedicated to disseminating project outcomes and findings to a wide audience. During this period, one peer-reviewed paper was published in a reputable journal, and another was presented at the SPE Annual Technical Conference and Exhibition. Additionally, three articles have been submitted to journals, with three more under internal review. Furthermore, three additional articles are currently being prepared for submission. These papers provide detailed descriptions of the research methodology, results, and their broader significance.
  - Conferences and Workshops: The project team prepared and presented a paper at the SPE Annual Technical Conference and Exhibition, held from September 23 to September 25, 2024. Also during this reporting period, Jeffrey Luo, the Technical Task Initiator (TTI), visited the experimental facility. Three technical presentations and one progress update were conducted, providing the TTI with a detailed tour of the experimental setup and a comprehensive explanation of the ongoing work.

(b) Financial Summary

- Federal Cost Activities: **Table 1** shows the expenses incurred in different budget categories as of September 30, 2024.

  - PI/Co-PIs/students involvement: The total yearly expenditure on salaries and wages amounts to $142,028, covering the graduate research assistant salaries and the PI's summer salary. To date, four graduate research assistants have been actively involved in various tasks related to the project. In the current reporting year, $56,490 has been allocated to tuition support for these graduate students. Additionally, we have hired an undergraduate student to assist with experimental work.

  - Materials purchased/travel/contractual (consultants/subcontractors): The total expenditure for supplies is $6,444, which includes costs for test materials such as hydrogen, natural gas, and nitrogen, as well as miscellaneous supplies necessary for conducting experiments. The total expenditure for fabricated equipment amounts to $46,554, covering items purchased to modify the experimental setups. These items include specimen holders and grips, hydrogen resistance strain gauges, tubing and fittings for high-pressure gas lines, measuring instruments, structural components for constructing the experimental setups, and heating jackets. Additional funds are needed in the other direct costs budget to cover charges related to specimen manufacturing and testing services.

Table 1: Federal cost by budget category for Year 1

No.	Budget Categories	Expenses
1	Salary & Wages	$ 142,028
2	Fringe Benefits	$ 29,453
3	Supplies	$ 6,444
4	Travel - Domestic	$ 3,213
5	Other - specimen manufacturing and testing services	$ 41,412
6	Equipment	$ 46,554
7	Tuition	$ 56,490
8	F&A 55%	$ 122,402
	Total	$ 447,996

- Cost Share Activities:

  - Cost share contribution: **Table 2** summarizes the University of Oklahoma's (OU) cost share during the reporting period, broken down by budget category. In Year 2, OU contributed a total of $98,836, which includes personnel salaries ($48,750), fringe benefits ($15,015), and overhead costs ($35,071). As part of this cost-sharing effort, the PI and Co-PI were actively involved in various research and development activities, including supervising research assistants and technical personnel, conducting hydrogen embrittlement research, and maintaining the operation of the experimental setups.

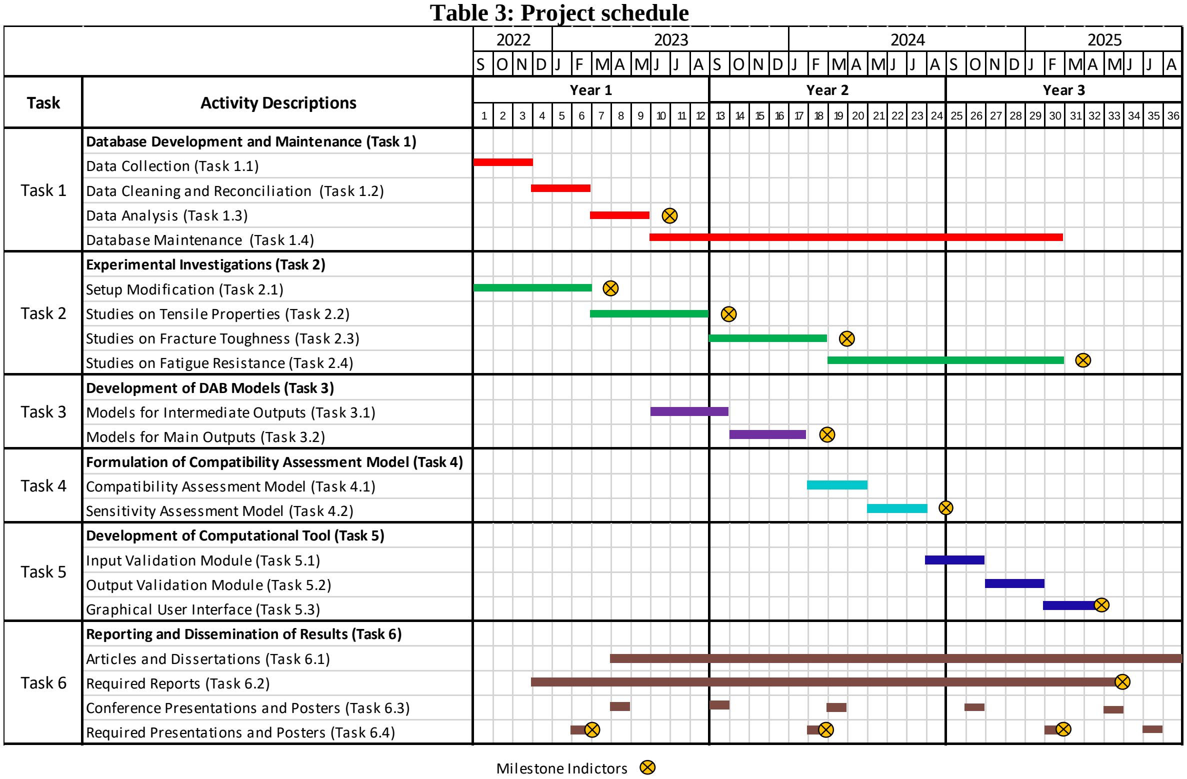
Table 2: OU cost share for Year 1

No.	Budget Categories	Year 2
1	Salary & Wages	$ 48,750
2	Fringe Benefits	$ 15,015
3	Supplies	$ -
4	Travel - Domestic	$ -
5	Other	$ -
6	Equipment	$ -
7	Tuition	$ -
8	F&A 55%	$ 35,071
	Total	$ 98,836

(c) Project Schedule Update

- Project Schedule: **Table 3** provides a comprehensive outline of the project tasks and milestones. We are proceeding as planned, with steady progress on all theoretical and modeling tasks, as well as the development of hydrogen embrittlement assessment tool that predict the lifespan of a pipeline, ensuring the project stays on schedule.

Table 3: Project schedule



(d) Status Update of the 8th Quarter Technical Activities

- <u>Task 1.4: Database Maintenance</u>

Our machine-learning models are regularly maintained to improve their accuracy and performance. Beginning in Quarter 4, we initiated continuous database maintenance, incorporating several newly published datasets. The database is steadily expanding with the addition of original experimental data, including tensile and fracture toughness measurements. As part of this maintenance process, the team has thoroughly examined and cleaned the database to correct any errors or inconsistencies. Additional tests have been conducted to ensure data accuracy, further validating the dataset.

- Task 2.3: Studies on Fracture Toughness

The fracture toughness testing task was successfully completed during this reporting period. A dedicated pre-cracking setup was constructed for this purpose (**Fig. 1**), designed with adjustable load ratio and frequency capabilities. The pre-cracking process was monitored using optical microscopy to ensure precision and accuracy. Following pre-cracking, the specimens were subjected to fracture toughness testing under controlled hydrogen environment conditions. A total of 100 samples, representing three distinct materials (X52, X60, X70), were tested in hydrogen environments with varying natural gas concentrations, temperatures, and oxygen concentration levels.

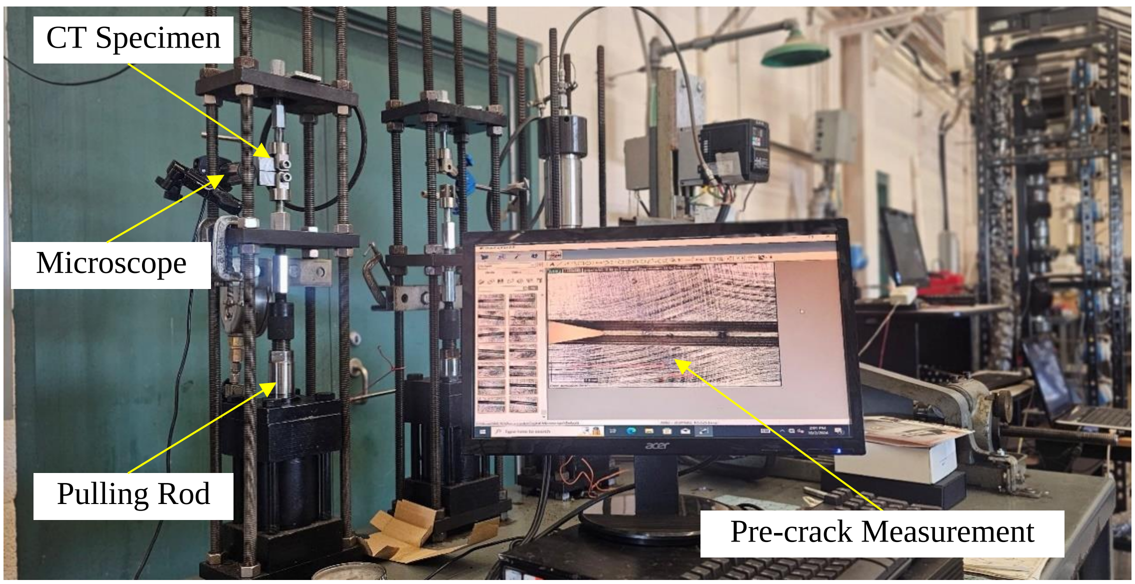


Fig. 1 Pre-cracking setup for fracture toughness test of CT sample

- Task 2.4: Studies on Fatigue Resistance

During this period, experimental studies on fatigue resistance commenced. The study is conducted using CT specimens that are pre-cracked. Fatigue crack growth under hydrogen-assisted conditions is measured in an autoclave under cyclic loading, within a controlled environment of hydrogen and natural gas blends. A total of 150 specimens are prepared by sectioning vintage pipeline material, which are preserved by immersing them in oil and sealing them in vacuum-sealed bags. According to the original project plan, we aim to complete these experiments over the next two quarters. Experiments will be conducted with varying stress intensity ranges, hydrogen concentrations, temperatures, and oxygen concentrations to determine the influence of these parameters on the fatigue crack growth rates of pipeline materials (X52, X60, and X70).

- Task 3.2: Model for Main output

  Our research team has developed two advanced machine-learning models designed to predict critical outputs for the hydrogen pipeline compatibility study: hydrogen-assisted fatigue crack growth rate and fracture toughness. These models incorporate a wide range of chemical, mechanical, and environmental variables to generate precise predictions. Both the fatigue crack growth and fracture toughness models utilize data sourced from literature and experimental findings to predict crack propagation in hydrogen-embrittled pipeline materials.

  To improve the models' predictive accuracy, we continuously update them with new experimental data, including critical stress intensity factor and fatigue crack growth rate measurements obtained in high-pressure hydrogen environments. The integration of these experimental findings into the machine-learning framework enables more reliable predictions of material behavior, aiding in the optimization of operating parameters to minimize the effects of hydrogen embrittlement. Additionally, the models support the selection of pipeline materials best suited for hydrogen transport applications.

  These models address the complex challenges posed by hydrogen embrittlement and contribute to the development of robust computational tools for predicting material degradation in hydrogen-rich environments, facilitating more effective compatibility assessments.

- Task 4: Formulation of Compatibility and sensitivity Assessment Models

  Research team members developed compatibility and sensitivity assessment models (Task 4.1 and Task 4.2) to assess hydrogen embrittlement's impact on fatigue crack resistance in materials during this reporting period. The model utilizes ASME B31.12 guidelines and accounts for factors such as initial crack size, operating pressure range, load fluctuation frequency, temperature, and material type and gas composition in the evaluation of fatigue crack growth rate. By simulating real-world conditions, including pressure variations and pipeline defects, the model estimates how long a pipeline can operate safely before requiring maintenance or facing crack related failure. This ensures that hydrogen pipelines are designed and maintained with high safety standards, supporting reliable hydrogen transport.

- Task 5: Development of Computational Tool

  Our research team began developing a user-friendly computational tool that allows users to input data, which runs both intermediate and main models in the background and generates a final assessment. The tool will assess compatibility, perform sensitivity analysis across various operating conditions, and present results with clear explanations and visualizations, enabling users to easily interpret the findings and make informed decisions.

Section B: Detailed Technical Results in the Report Period

1. Background and Objectives in the 2nd Annual Report Period

Background

This study aims to investigate Hydrogen Embrittlement (HE) in pipeline materials and assess their suitability for transporting both blended and pure hydrogen gases. Due to the complex nature of hydrogen permeation and the various factors influencing embrittlement, existing HE models have limited applicability. Additionally, there is insufficient field data to fully evaluate these models under diverse conditions. However, preliminary research indicates that Data Analytics Based (DAB) models could effectively predict HE's impact on mechanical properties. With a properly formulated and extensively tested DAB model, it may be possible to predict HE failure across different conditions and materials.

As part of this project, DAB modeling techniques are being developed to create a comprehensive compatibility assessment model for evaluating the use of existing pipelines for hydrogen and blended gas transportation. A queryable database has been established using publicly available experimental data to support the development of the DAB model. This database will be further expanded through ongoing experiments. Various data analytics methods have been reviewed and those most suitable for predicting HE have been selected. The compatibility assessment model will integrate methods that demonstrate reliable performance. The project's primary deliverable will be a computational tool based on this model, enabling the assessment of a pipeline's suitability for hydrogen or blended gas transport. Additionally, the tool will help determine if HE-inhibiting impurities are needed or if adjustments to gas processing operations are required.

Objectives in the 2nd Annual Report Period

The 3-year (36-month) project, which commenced on September 30th, 2022, and will conclude on September 29th, 2025, has set several key objectives for the second year. These include: i) improving and maintaining the database by conducting experiments and incorporating published results (Task 1.4); ii) performing experimental studies to assess the impact of hydrogen embrittlement on the fracture toughness of pipeline materials (Task 2.3); iii) conducting experiments to evaluate the fatigue resistance of pipeline materials in hydrogen-rich environments (Task 2.4); iv) developing key data analytics-based models required for the formulation of the compatibility assessment model (Task 3.2); v) creating the compatibility assessment model to predict the life expectancy of pipeline materials affected by hydrogen embrittlement (Task 4.1); and vi) developing a sensitivity model to evaluate the impact of varying parameters on hydrogen embrittlement.

2. Experimental Program in the 2nd Annual Report Period

Experimental Design

The existing test setup, consisting of three autoclaves, has been modified to conduct hydrogen embrittlement (HE) experiments on various pipeline materials. Two newly built autoclaves, each with a 3.1-liter capacity, have been added to facilitate in-situ HE testing on pipeline steel (**Fig. 2**). These new autoclaves are designed to accommodate different specimen holders and clip-gauge assemblies. The autoclaves are equipped with jackets to enable temperature control through the circulation of a glycol-based heat transfer fluid during experiments. The fluid's temperature is regulated by a high-capacity chiller and a 6-kW electric heater. During the tests, an axial force is applied to the specimen using a hydraulic cylinder mounted below the setup to stretch the pulling rod.

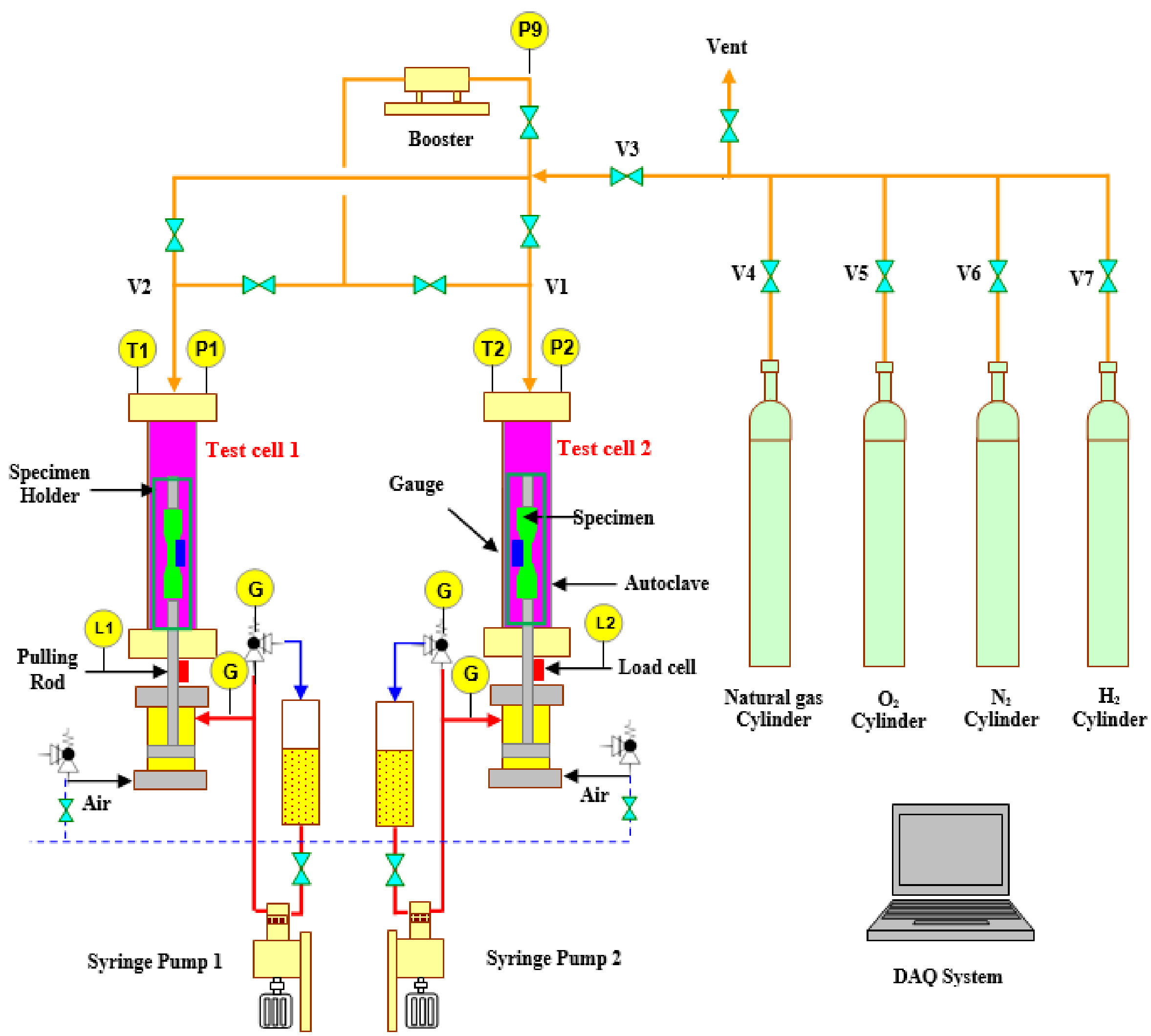


Fig. 2 Simplified schematic of test setup

Test Procedure

To perform the experimental investigation, commonly used grades of vintage pipeline steel (X52, X60, and X70) were sourced from the Pipeline Research Council International. The pipes were of sufficient thickness to produce both flat and cylindrical specimens. A total of 150 specimens were fabricated, with 50 specimens for each material grade. These specimens are stored in a dry vacuum environment to prevent surface corrosion. Each specimen is clearly marked to identify its material grade and avoid any potential errors during measurement and reporting.

The experimental testing process consists of several key steps, including pre-cracking, cleaning and preparation, specimen placement in the autoclave, purging the autoclave, adjusting the autoclave temperature, pressurizing the cell, hydrogen charging, applying the load, measuring strain, and post-test evaluation. These steps are detailed below:

1. **Pre-cracking**: Fatigue pre-cracking is required for compact tension (CT) specimens used in fracture toughness and fatigue strength testing. The pre-cracking process is conducted under atmospheric conditions by applying a fluctuating load at a constant load ratio while monitoring crack opening displacement using an optical microscope.
2. **Cleaning and Preparation**: Specimens must be cleaned and prepared with uniform surface smoothness to ensure consistent measurements. Standard surface finishes are applied, and the specimens are stored in vacuum-sealed containers. Before testing, each specimen is degreased and cleaned with acetone.
3. **Specimen Placement in the Autoclave**: The autoclave's lid is removed, and the specimen holder is raised using a hydraulic cylinder. The cylinder is pneumatically actuated to lift the holder. The specimen is then positioned in the holder, and a clip gauge is attached. The holder components, including the top disk and thumbscrews, are assembled, and the holder is lowered back into place by retracting the hydraulic cylinder. The gas injection and distribution tubes are connected to the autoclave, and the lid is secured. Gas inlet and outlet lines are connected, and a thermocouple is inserted to monitor temperature.
4. **Autoclave Purging and Temperature Adjustment**: The autoclave is purged of air, and its temperature is adjusted simultaneously. A heating or cooling medium is circulated through the autoclave jacket to control the internal temperature. Purging is done stepwise using the test gases (either natural gas or hydrogen) while monitoring the oxygen concentration using a gas analyzer (**Fig. 3**). Temperature and oxygen content are monitored during this process, and purging is completed when the desired temperature and oxygen levels are reached.
5. **Cell Pressurization**: The autoclave is pressurized stepwise to ensure accurate gas composition. Gases are injected according to their molar fraction, starting with the gas of the lowest molar fraction and ending with the one of the highest.
6. **Hydrogen Charging**: After pressurization, the specimen is aged in the autoclave for one hour. Temperature is controlled automatically, and pressure is manually adjusted to maintain the desired levels throughout the charging process.
7. **Testing**: After hydrogen charging, the desired load is applied to the specimen, and strain is measured as a function of time. The experiment continues until specimen failure,

indicated by an abrupt load drop.

8. **Specimen Recovery**: After the test, the autoclave is depressurized, and the specimen is retrieved for further examination.
9. **Microscopic Evaluation**: The specimen is examined under an optical microscope to assess crack characteristics, failure mode, and crack size (**Fig 4**).

This comprehensive testing process ensures precise evaluation of the fracture toughness and fatigue resistance of the pipeline materials under hydrogen-assisted conditions.

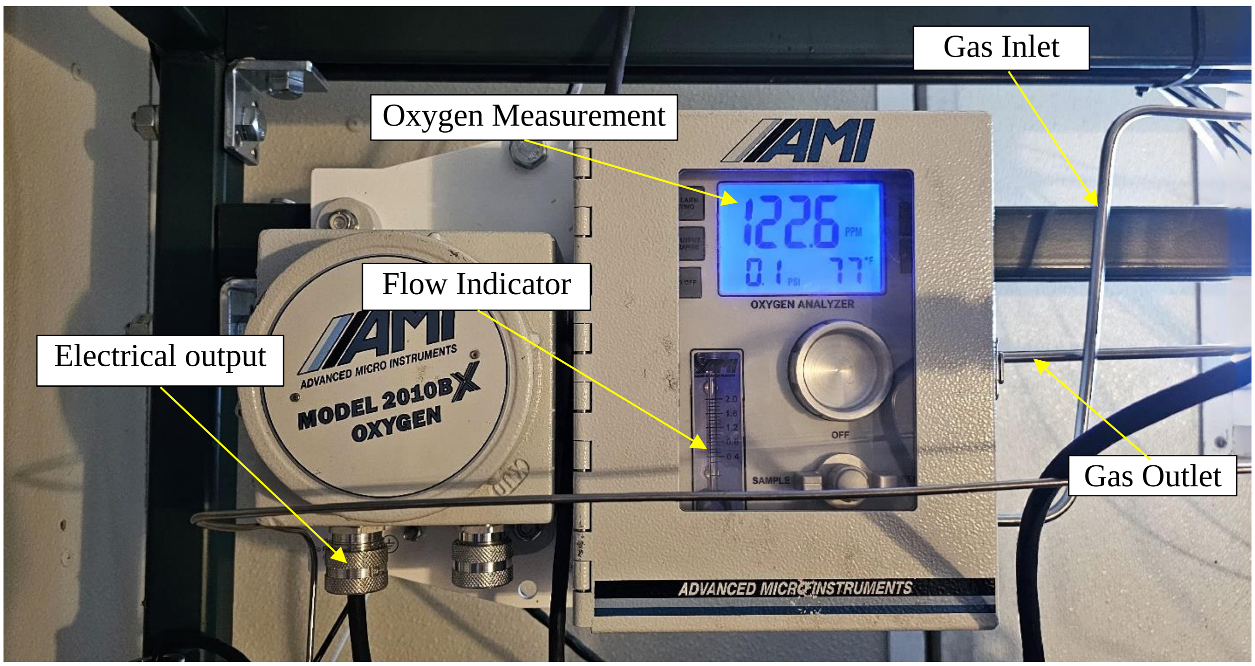


Fig. 3 Gas Analyzer setup for oxygen concentration measurement

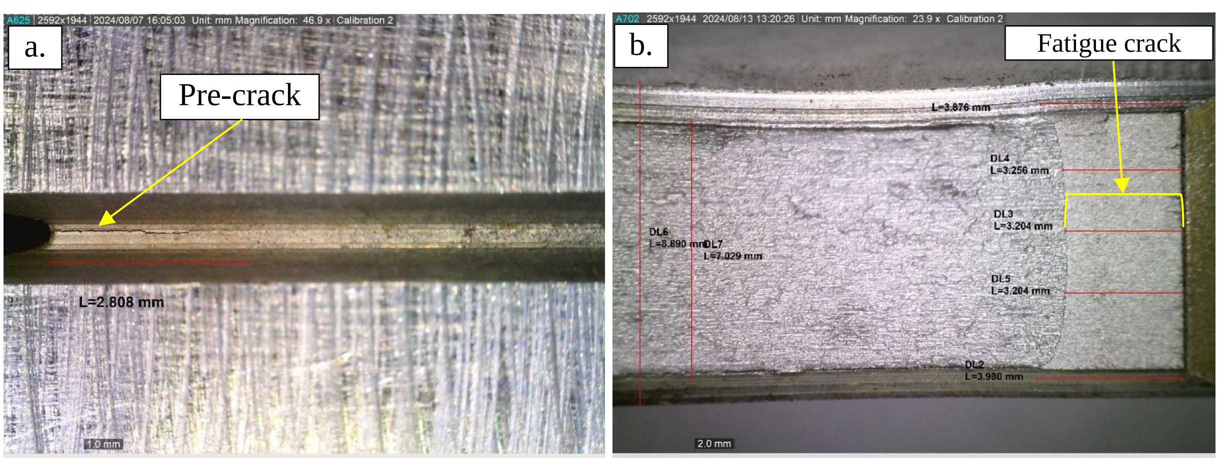


Fig. 4 Microscopic measurement of (a) pre-crack, and (b) fractured surface in an X60 specimen.

3. Results and Discussions

Task 1: Database Development and Maintenance

- Task 1.4: Database Maintenance

To enhance the performance and accuracy of our machine learning models, we continuously update and maintain the database. As new experimental data is generated throughout the project and additional data from other hydrogen embrittlement (HE) studies is incorporated, the database will be expanded. By the end of this period, we completed tensile and fracture toughness testing of pipeline materials, and this data has been carefully examined and added to the database. We also perform periodic data cleaning to identify and correct any inaccuracies or inconsistencies. The database revision history is carefully documented after each maintenance cycle. To prevent data loss, corruption, or deletion, backup copies are regularly created to ensure the restoration of the original data if needed.

Task 2: Experimental Investigations

The experimental work consists of four subtasks: setup modification (Task 2.1), studies on tensile properties (Task 2.2), studies on fracture toughness (Task 2.3), and studies on fatigue resistance (Task 2.4), as outlined in Table 3. Tasks 2.1 and 2.2 were completed during the first reporting period and reported accordingly. In the current reporting period (2nd cycle), studies on fracture toughness have been completed (Task 2.3), and studies on fatigue resistance (Task 2.4) have commenced.

- Task 2.3: Studies on Fracture Toughness

This task aims to investigate the impact of hydrogen embrittlement (HE) on the fracture toughness of pipeline materials. To replicate actual pipeline conditions, specimens are collected from vintage pipeline materials. The specimens are cut and prepared as compact tensile specimens in accordance with ASTM standards. Pre-cracking is performed using variable cyclic loading and frequency. Each specimen is pre-cracked within a specified range (2.5–3.1 mm), and an electronic microscope is used to measure and monitor the pre-crack size during the process. Pre-cracking is conducted in atmospheric conditions.

After pre-cracking, the specimens are cleaned with acetone and fracture toughness tests are conducted in high-pressure hydrogen gas environments, or in hydrogen blended with natural gas, with hydrogen concentrations ranging from 0 to 100%. To assess the effects of oxygen concentration and temperature on HE and fracture toughness, tests are performed under varying oxygen levels (0–1000 ppm) and temperatures ranging from 50 to 122°F. The fracture toughness tests are carried out at a constant loading rate until the specimens completely fracture. The maximum load prior to failure is recorded, and the crack size is measured using an electronic microscope. Finally, the fracture toughness for each specimen is calculated. **Fig. 5** illustrates the load line displacement measurement of the CT specimen under a constant loading rate during the fracture toughness test in a hydrogen environment. Initially, the load increases steadily as the material resists fracture. Upon reaching the critical stress intensity conditions, which corresponds to the peak fracture resistance, the material begins to show fracture failure. As a result, the load decreases, indicating that the material’s capacity to bear the load has been compromised, causing accelerated crack growth.

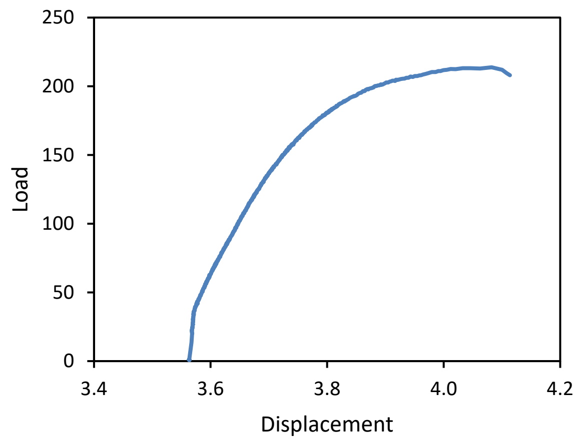


Fig. 5 Increment of displacement due to applied load

The experimental results for fracture toughness of the three pipeline materials (X52, X60, and X70) across varying hydrogen concentrations reveal several important trends **(Fig. 6)**. As the hydrogen content increases, the fracture toughness consistently decreases for all materials, confirming the embrittlement effect of hydrogen. The data shows that the X52 material is the most susceptible, with a significant drop in fracture toughness even at low hydrogen concentrations. In contrast, X70 exhibits the highest resistance to hydrogen embrittlement, maintaining relatively high fracture toughness values across the entire range of hydrogen content.

A key observation is that the most significant drop in fracture toughness occurs at lower hydrogen concentrations, particularly around 6.25%. This sharp reduction suggests that even small amounts of hydrogen can significantly degrade the material's toughness by saturating the crack tip and accelerating embrittlement. Beyond this point, the rate of fracture toughness reduction plateaus for all materials, indicating that additional hydrogen has a diminishing effect on further toughness degradation. This plateau suggests that the critical mechanisms responsible for hydrogen-induced embrittlement are fully activated at low hydrogen levels, and increasing the hydrogen content beyond this threshold does not significantly worsen the material's performance.

The differing behaviors between the materials also provide insights into their suitability for hydrogen transport. X70, with its higher fracture toughness and slower rate of decline, is the most resilient to hydrogen exposure, making it more suitable for applications where higher hydrogen concentrations are expected. Conversely, X52, with its more pronounced susceptibility, may be less suited for such conditions, requiring more protection or adjustments in operating conditions to ensure structural integrity. This data is crucial for developing models that predict pipeline performance under hydrogen-rich environments, aiding in the selection of appropriate materials based on expected hydrogen exposure.

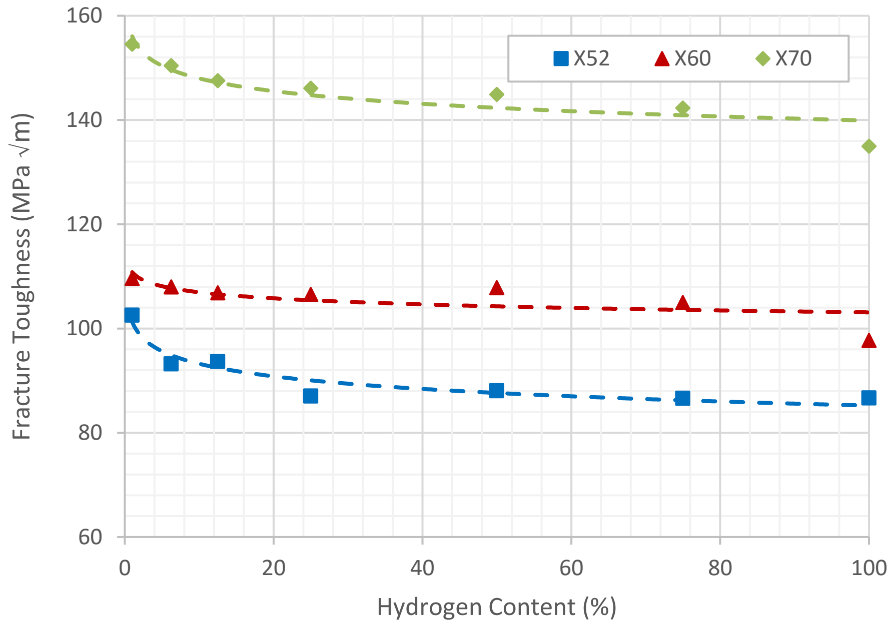


Fig. 6 Fracture Toughness of Pipeline Materials at different hydrogen concentrations.

- <u>Task 2.4: Studies on Fatigue Resistance</u>

This task aims to investigate the impact of hydrogen embrittlement (HE) on the fatigue resistance and fatigue crack growth behavior of steel used in pipelines. For these experiments, compact tension (CT) specimens are prepared from vintage pipeline materials. Before conducting the main fatigue tests, the specimens are pre-cracked using cyclic loading in atmospheric conditions to simulate natural fatigue crack formation. Once pre-cracked, the main fatigue experiments are conducted in a hydrogen-rich environment to evaluate the influence of hydrogen on crack growth and material degradation under operational conditions. This task is expected to be completed in the middle of the next reporting period.

Task 3: Development of DAB Models

This task aims to establish data analytics-based (DAB) models for main and intermediate outputs. It consists of model development for the intermediate (Task 3.1) and main (Task 3.2) outputs.

- <u>Task 3.2: Models for Main Outputs</u>

Fatigue Crack Growth Rate Prediction:

A detailed database has been compiled through an extensive review of fatigue test results from key studies [2-10]. The database includes fatigue data for 16 distinct types of carbon steel, comprising 3402 individual data points characterized by 26 different testing parameters. To support a novel machine learning approach, the reduction of area has been added as a new attribute, increasing the total number of attributes to 27. The database captures steel composition, mechanical properties, and testing environment details. In addition to the 16 carbon steels, various welded versions and specific condition variations are also included, bringing the total to 36 different sample classes. The focus of the data collection was limited to carbon and low-alloy steels, which are widely used and critically important for pipeline transport applications [1]. The fatigue tests were conducted in environments including air, helium, and high-pressure hydrogen gas, while tests involving electrochemical charging mechanisms were intentionally excluded, as they do not represent the conditions typically found in pipeline transport. A detailed summary of the database is provided in **Table 4**.

Table 4— Overview of the Dataset for Developing the ML Model on Fatigue Crack Growth Rate

Categories	Parameters	Data Range (Minimum/Maximum)	Mean	Standard Deviation	Units
Chemical Properties	Iron (Fe)	92.73/99.31	97.99	0.992	%
	Carbon (C)	0.03/0.85	0.162	0.152	%
	Manganese (Mn)	0.30/1.87	1.068	0.383	%
	Phosphorous (P)	0.001/0.02	0.011	0.0042	%
	Sulfur (S)	0.00/0.13	0.017	0.022	%
	Silicon (Si)	0.001/0.73	0.170	0.129	%
	Chromium (Cr)	0.00/1.68	0.143	0.288	%
	Nickel (Ni)	0.001/4.96	0.218	0.645	%
	Molybdenum (Mo)	0.001/0.43	0.072	0.128	%
	Aluminum (Al)	0.001/0.42	0.022	0.066	%
	Copper (Cu)	0.001/0.31	0.092	0.103	%
Mechanical Properties	Yield Strength (Sy)	207/965	464.018	134.773	MPa
	Ultimate Strength (UTS)	379/ 1020	586.203	130.922	MPa
	Stress Intensity Factor (ΔK)	3.890/145.593	17.063	10.707	MPa.m$^{1/2}$
	FCG Rate (da/dN)	7.7E-07/ 0.064	0.00093	0.0029	mm/cycle
	Reduction of Area (RA)	23.88/77.23	54.89	10.102	%
Test Conditions	Hydrogen Partial Pressure	0.00/103.55	15.481	23.93	MPa
	Load Ratio	0.007/1.0	0.372	0.220	
	Frequency	0.001/10	1.475	18.626	Hz
	Stress Intensity Factor (ΔK)	3.890/145.593	17.063	10.707	MPa.m$^{1/2}$
	N_2 Content	0.00/1.00	-	-	-
	O_2 Content	0.00/1.00	-	-	-
	CO_2 Content	0.00/1.00	-	-	-
	Heat Treatment	Different heating treatment	-	-	-
	Product Form	Various sources*	-	-	-

This study investigates the development of machine learning (ML) models for predicting fatigue crack growth (FCG) rates using two different approaches: a traditional method and an innovative strategy. The traditional approach, or conventional method, relies solely on data from hydrogen-assisted fatigue tests to predict FCG rates. In contrast, the innovative strategy uses a dual-model technique. It first applies fatigue test data to a model previously developed from tensile test data to estimate the reduction of area (RA). The predicted RA values are then used as inputs in a secondary model, trained with fatigue test data, to predict FCG rates.

Both approaches are tested using a variety of ML algorithms, including Decision Trees, Random Forests, Gradient Boosting, Extreme Gradient Boosting, Adaptive Boosting, Categorical Boosting, and K-Nearest Neighbors. The performance of the models is evaluated using metrics such as coefficients of determination, mean squared error (MSE), root mean squared error (RMSE), mean absolute error (MAE) **(Table 5)**, and the relative ranking of feature importance **(Table 6)**. After this comparative analysis, the CatBoost model emerged as the best-performing model, demonstrating the closest results to the calculated averages of these performance metrics. In our research, the CatBoost model was used to explore the fatigue behavior of pipeline materials. We partitioned the dataset into 80% for training and 20% for testing to assess the model's predictive performance. The primary goal of using CatBoost in this study was to accurately predict FCG rates in specific types of pipeline steels. These predictions were based on the analysis of key factors such as hydrogen pressure in the pipeline, the chemical and mechanical properties of the steel, and the heat treatment conditions. This approach not only improves the understanding of the factors influencing pipeline material durability but also provides valuable insights into the potential for material failure, contributing to advancements in materials engineering and pipeline safety.

Table 5 Summary of FCG ML model performance metrics

Model Name	Coefficient of Determination		MSE	RMSE	MAE
	Training R^2	Test R^2			
KNN	0.8028	0.81	1.99E-06	0.0013	0.00033
RF	0.8025	0.8007	2.00E-06	0.0014	0.00033
DT	0.76	0.75	2.64E-06	0.0016	0.00036
XGBoost	0.8878	0.8766	1.30E-06	0.0011	0.00034
AdaBoost	0.8252	0.7903	2.20E-06	0.0015	0.00031
GB	0.8819	0.7982	2.12E-06	0.0015	0.00032
CatBoost	0.9215	0.8542	1.49E-06	0.0012	0.00031
	0.9215	0.8766	1.30E-06	0.0011	0.00031
	Max		Min		

Table 6 Summary of FCG ML model feature importance rankings

Model Name	Feature Importances Ranking																	
	Fe	C	Si	S	P	Al	Mn	Cr	Ni	Mo	ΔK	H2 Pressure	UTS	LR	Frequency	PF	O2 ppm	HT*
KNN	11	17	10	14	4	16	13	8	18	15	1	7	12	3	2	5	9	6
RF	6	7	13	16	15	11	8	10	12	9	1	2	3	4	5	18	17	14
DT	18	9	8	4	7	16	3	14	12	17	1	2	6	5	11	15	13	10
XGBoost	18	1	15	12	13	17	10	16	6	14	3	4	11	8	2	7	5	9
AdaBoost	8	13	11	12	10	16	7	5	14	15	1	2	3	4	6	17	18	9
GB	13	7	4	5	12	8	14	16	3	11	1	2	9	6	10	17	15	18
CatBoost	9	17	13	15	7	12	6	10	11	14	1	2	4	3	5	18	8	16
Average	11.85	10.14	10.57	11.14	9.71	13.71	8.71	11.29	10.86	13.57	1.29	3	6.86	4.71	5.86	13.86	12.14	11.72
Rank	14	8	9	12	7	17	6	11	10	16	1	2	5	3	4	18	15	13

Fracture Toughness Prediction:

Hydrogen embrittlement significantly reduces the mechanical performance of these steels, compromising their fracture toughness, which is critical for maintaining the integrity of pipelines and storage facilities in hydrogen-based infrastructure. The study seeks to advance the understanding of HE by using machine learning (ML) models to predict the fracture toughness of these steels, thereby providing a framework for developing HE-resistant materials. The primary goal of this research is to create a predictive model that accurately estimates the fracture toughness of low-carbon and low-alloy steels exposed to hydrogen-rich environments. This model helps in identifying the critical factors affecting hydrogen embrittlement and suggests potential material type and operating conditions to improve steel resistance to embrittlement. The findings can be used to inform safer design and maintenance protocols for hydrogen storage and transport infrastructure.

The dataset used in the study was compiled from a range of sources, including peer-reviewed publications, technical reports, and conference proceedings [11-27]. It primarily contained experimental data from fracture toughness tests conducted on low-carbon and low-alloy steels exposed to various environmental conditions, including air, natural gas, high-pressure hydrogen gas, and gaseous impurities such as oxygen and carbon dioxide. After rigorous data cleaning and preprocessing, 180 data points were selected from the initial 210, covering 18 key features, including chemical properties (iron, carbon, manganese, phosphorus, silicon, etc.), mechanical properties (yield strength, ultimate tensile strength), and test conditions (hydrogen pressure, displacement rate) (**Table 7**). To ensure model accuracy, the dataset underwent a process of feature selection, which narrowed the focus to 13 parameters most relevant to predicting fracture toughness, such as hydrogen partial pressure, yield strength, and the concentrations of minor alloying elements.

Table 7 Overview of the Dataset for Developing the ML Model on Fracture Toughness

Categories	Parameters	Range (Min/Max)	Units
Chemical Properties	Iron (Fe)	93.86/99.604	%
	Carbon (C)	0.03/0.49	%
	Manganese (Mn)	0.04/1.72	%
	Phosphorous (P)	0.0/0.033	%
	Sulfur (S)	0.00/0.035	%
	Silicon (Si)	0.014/1.08	%
	Copper (Cu)	0.00/0.31	%
	Other*	0.00/5.36	%
	Aluminum (Al)	0.00/0.42	%
Mechanical Properties and Test Conditions	Yield Strength (Su)	280/1086	MPa
	Ultimate Strength (Su)	415/ 1198	MPa
	Fracture toughness (FT)	20/393	MPa√m
	Hydrogen Partial Pressure	0.10/97.00	MPa
	Displacement Rate	1.272/0.001	mm/min
	Heat Treatment	Different heating treatment	-
	Product Form	Diverse product⁺	-
	0xygen	5/100	ppm
	Carbon dioxide	0/0.69	Mpa

Seven machine learning models were initially evaluated for their ability to predict fracture toughness based on material properties and environmental factors. These models were: K-Nearest Neighbors (KNN), Random Forest (RF), Gradient Boosting (GB), Decision Tree (DT), Support Vector Machine (SVM), Artificial Neural Networks (ANN), and CatBoost. After initial testing, the top four models (KNN, RF, GB, and DT) were selected for detailed evaluation based on their performance in accurately predicting fracture toughness **(Table 8)**. These models were chosen because they consistently identified the key predictive factors of fracture toughness and exhibited minimal overfitting. Among these models, K-Nearest Neighbors (KNN) emerged as the most effective. KNN's strong performance in both training and testing datasets made it the most reliable for predicting the effects of hydrogen embrittlement on fracture toughness.

Table 8 Summary of fracture toughness ML model performance metrics

Model	Coefficient of Determination			RMSE	MAE
	Train R^2	Test R^2	ΔR^2		
RF	0.77	0.76	0.01	41.41	29.06
DT	0.78	0.73	0.05	44.43	33.99
GBOOST	0.82	0.80	0.02	38.41	28.72
KNN	0.85	0.84	0.01	33.57	25.95
max	0.85	0.84	0.05	46.73	33.99
min	0.77	0.70	0.01	33.57	25.95

The study found that hydrogen pressure is the most influential factor affecting fracture toughness, with a significant decrease observed at pressures below 8 MPa and a sharp decline at 6.9 MPa, after which the effect stabilizes, indicating a saturation point. Higher yield strength materials were more susceptible to hydrogen embrittlement, demonstrating a trade-off between strength and toughness, as increased strength led to reduced fracture resistance. Additionally, the displacement rate during testing played a crucial role, with higher rates leading to improved toughness, particularly in the presence of hydrogen. Alloying elements, such as carbon, phosphorus, nickel, and others, also significantly influenced toughness, accounting for around 10% of the overall impact, while small amounts of oxygen (5–20 ppm) were found to mitigate hydrogen embrittlement. Carbon and phosphorus were identified as the most critical elements in reducing toughness, with carbon increasing hardness and brittleness, and phosphorus weakening interfaces, making the material more prone to crack propagation.

Task 4: Formulation of Compatibility Assessment Model

The goal of this task is to create a model that predicts the life expectancy of pipeline materials in hydrogen-rich environments. It involves developing two key components: the Compatibility Assessment Model (Task 4.1) and the Sensitivity Assessment Model (Task 4.2).

- <u>Task 4.1: Compatibility Assessment Model</u>

The Compatibility Assessment Model evaluates the suitability of existing pipelines for hydrogen transport by assessing their vulnerability to hydrogen embrittlement and fatigue crack growth. Hydrogen transport poses unique challenges as it can cause embrittlement in pipeline steels, which significantly reduces the material's ductility and fracture toughness, leading to crack formation and growth. The model focuses on assessing the material susceptibility to hydrogen embrittlement, with specific attention to different grades of pipeline steels.

A key aspect of the model is the use of main model output (fatigue crack growth prediction and fracture toughness prediction) to predict the life expectancy under hydrogen service conditions. The cyclic loading condition of hydrogen pipelines is also accounted for. The cyclic loading accelerates crack growth in the presence of hydrogen, making it essential to assess the pipeline's ability to withstand repeated pressure variations without failure. The model defines a critical crack size that depends on the pipeline's wall thickness and material properties. If the crack grows beyond this size, the pipeline is considered at risk of failure. Additionally, the model incorporates the threshold stress intensity factor for hydrogen-assisted cracking to ensure that the pipeline does not exceed safe operational limits. By simulating crack growth over multiple load cycles, the model provides an estimate of the number of allowable load cycles before the pipeline fails. The model's predictions have been validated against existing studies (**Fig. 7**), offering deeper insights into pipeline durability in hydrogen environments. This allows for the prediction of the pipeline's operational lifespan under hydrogen service conditions. The Compatibility Assessment Model acts as a predictive tool to assess the structural integrity of pipelines for hydrogen transport. By combining material susceptibility, crack growth prediction, and cyclic loading conditions, the model ensures that hydrogen pipelines can endure the stresses imposed during operation. This is vital in determining whether existing natural gas pipelines can be safely requalified for hydrogen use or if new hydrogen-specific pipelines are required to meet safety standards.

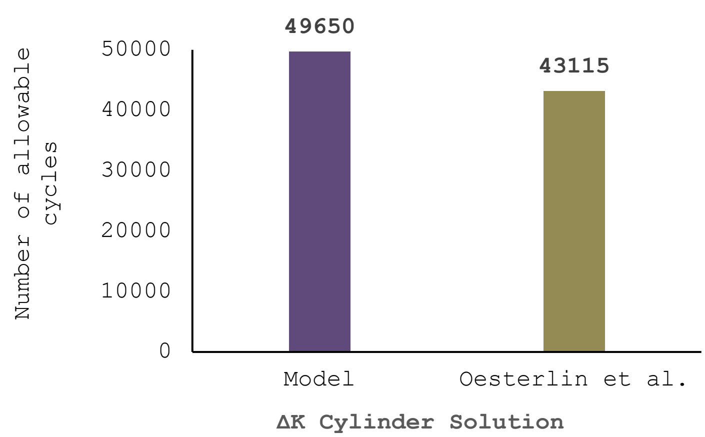


Fig. 7 Comparison of allowable cycles for ΔK Cylinder Solution with Literature using B31.12 and API 579-1.

The developed lifetime prediction model was compared with existing models from Oesterlin (2024) [28] and Fischer (2023) [29], focusing on allowable fatigue cycles under varying initial crack depths and pressure ratios (Fig. 7). The results indicate that the developed model predicts a higher number of allowable cycles (49,650) compared to Oesterlin's model (43,115), suggesting a slightly less conservative approach. The analysis further shows that as the initial crack depth increases, the allowable number of cycles decreases significantly. For instance, at 2.5% crack depth, the model predicts 267,725 cycles, while for 10% depth, the prediction drops to 7,850 cycles (**Fig. 8**). This reduction is consistent with the understanding that larger cracks result in higher stress intensities, accelerating crack growth. The differences between the models are primarily due to the use of different stress intensity factor solutions. Oesterlin's model employs solutions for surface-cracked plates, whereas the developed model uses a solution tailored for cylindrical structures with through-wall cracks. Additionally, at lower pressure ratios, the developed model predicts a substantially higher number of allowable cycles compared to the literature. However, for larger cracks and higher-pressure differentials, it shows more conservative predictions, indicating faster crack propagation. Overall, the developed model aligns closely with literature for smaller cracks but predicts fewer cycles for larger cracks, highlighting its greater sensitivity to initial crack size and pressure variations.

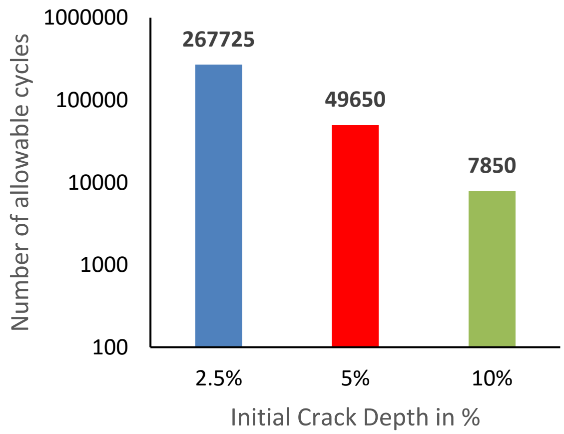


Fig. 8 Effect of Initial Crack Depth on the Predicted Allowable Cycles.

- <u>Task 4.2: Sensitivity Assessment Model</u>

Assessing the suitability of pipelines for transporting blended or pure hydrogen requires a comprehensive model that accounts for various input parameters. These inputs have varying degrees of influence on the overall outcome, with some playing a more significant role than others. Identifying the most critical parameters is essential for pipeline integrity managers to maintain them within acceptable thresholds, ensuring safe and reliable operation. Additionally, the model incorporates the effect of oxygen impurities, which is crucial for minimizing the risk of hydrogen embrittlement (HE).

To enhance the accuracy of the assessment, a sensitivity analysis is employed. This analysis begins by using baseline input values, followed by systematically varying each input while holding the others constant. The sensitivity of each parameter is measured by comparing the change in the primary output to the variation in the input value. This process allows for identifying the most influential factors, providing insights that can guide decision-making in maintaining pipeline integrity. We have already developed a sensitivity assessment model and ongoing refinement of the model is focused on improving its predictive capability for more reliable outcomes.

Task 5: Development of Computational Tool

This task focuses on the development of components of the tool including input validation module (Task 5.1), output validation module (Task 5.2), and Graphical User Interface (Task 5.3).

- Task 5.1 Input Validation Module

The input validation module is designed to prevent the tool from crashing due to invalid inputs or producing inaccurate predictions from out-of-range values. To address this, we are developing a validation system that verifies input validity, alerts the user when necessary, and specifies the acceptable input range.

4. Future work

In the third year, we will focus on several key activities, including conducting experiments, refining models, and developing a computational tool. The following research and development tasks are planned for the upcoming year:

- Continuing database enrichment and maintenance (Task 1.4),
- Performing experimental studies on the fatigue resistance of pipeline materials (Task 2.4),
- Further development of the compatibility assessment model (Task 4), and
- Developing a computational tool (Task 5), which includes the Input Validation Module (Task 5.1), Output Validation Module (Task 5.2), and Graphical User Interface (Task 5.3).

References

1. Ahmed, N., Aldaw, M., Ahmed, R., & Teodoriu, C. (2024). Modeling of necking area reduction of carbon steel in hydrogen environment using machine learning approach. *Engineering Failure Analysis, 156*, 107864. https://doi.org/10.1016/j.engfailanal.2023.107864
2. Nelson, H. (1976a). On the mechanism of hydrogen-enhanced crack growth in ferritic steels. In *Proceedings of the Second International Conference on Mechanical Behavior of Materials* (pp. 690-694). ASM, Metals Park, OH.
3. Nelson, H. G. (1976b). Hydrogen-induced slow crack growth of a plain carbon pipeline steel under conditions of cyclic loading. *Metallurgical Society of AIME*, United States.
4. Ronevich, J. A., Somerday, B. P., & Feng, Z. (2017). Hydrogen accelerated fatigue crack growth of friction stir welded X52 steel pipe. *International Journal of Hydrogen Energy, 42*(7), 4259-4268. https://doi.org/10.1016/j.ijhydene.2016.10.153
5. Ronevich, J. A., Somerday, B. P., & San Marchi, C. W. (2016). Effects of microstructure banding on hydrogen assisted fatigue crack growth in X65 pipeline steels. *International Journal of Fatigue, 82*, 497-504. https://doi.org/10.1016/j.ijfatigue.2015.09.004
6. San Marchi, C., et al. (2010). Fracture and fatigue of commercial grade API pipeline steels in gaseous hydrogen. https://doi.org/10.1115/PVP2010-25825
7. Somerday, B., San Marchi, C., & Nibur, K. (2013). Measurement of fatigue crack growth rates for SA-372 Gr. J steel in 100 MPa hydrogen gas following Article KD-10. https://doi.org/10.1115/PVP2013-97455
8. Wachob, H. F., & Nelson, H. G. (1980). Influence of microstructure on the fatigue crack growth of A516 in hydrogen. In I. M. Bernstein & A. W. Thompson (Eds.), *Proceedings of the third international conference on effect of hydrogen on behavior of materials* (pp. 703-711). The Metallurgical Society of AIME, Warrendale, PA.
9. Walter, R., & Chandler, W. (1973). Influence of gaseous hydrogen on metals final report. *NASA-CR-124410*, NASA, Marshall Space Flight Center, AL.
10. Walter, R., & Chandler, W. (1976). Cyclic-load crack growth in ASME SA-105 grade II steel in high-pressure hydrogen at ambient temperature. In T. M. S. of AIME (Ed.), *Effect of hydrogen on behavior of materials* (pp. 273-286). Warrendale, PA.
11. Peral, L. B., et al. (2019). Effects of hydrogen on the fracture toughness of CrMo and CrMoV steels quenched and tempered at different temperatures. *International Journal of Hydrogen Energy, 44*(7), 3953-3965.
12. Ronevich, J. A., et al. (2021). Hydrogen-assisted fracture resistance of pipeline welds in gaseous hydrogen. *International Journal of Hydrogen Energy, 46*(10), 7601-7614.
13. Ronevich, J., et al. (2018). Oxygen impurity effects on hydrogen assisted fatigue and fracture of X100 pipeline steel. In *ASME 2018 Pressure Vessels and Piping Conference*.
14. Nibur, K. A., et al. (2013). The relationship between crack-tip strain and subcritical cracking thresholds for steels in high-pressure hydrogen gas. *Metallurgical and Materials Transactions A, 44*, 248-269.
15. San Marchi, C., et al. (2011). Fracture resistance and fatigue crack growth of X80 pipeline steel in gaseous hydrogen. In *ASME 2011 Pressure Vessels and Piping Conference*.

16. Stalheim, D., et al. (2010). Microstructure and mechanical property performance of commercial grade API pipeline steels in high-pressure gaseous hydrogen. In *2010 8th International Pipeline Conference*.
17. Nishihara, Y., & Okano, H. (2023). Effect of high-pressure hydrogen gas on fatigue and fracture properties of API X65 linepipe steels. In *ASME 2023 Pressure Vessels & Piping Conference*.
18. San Marchi, C., et al. (2010). Fracture and fatigue of commercial grade API pipeline steels in gaseous hydrogen. In *ASME 2010 Pressure Vessels and Piping Division/K-PVP Conference*.
19. Hoover, W. R., et al. (1981). Hydrogen compatibility of structural materials for energy storage and transmission. *Final report*. United States.
20. Hoover, W. R., et al. (1980). Hydrogen compatibility of structural materials for energy storage and transmission. *Annual report, October 1, 1978-September 30, 1979*. United States.
21. Hoover, W. R. I., et al. (1980). Hydrogen compatibility of structural materials for energy storage and transmission. *Annual report*. University of North Texas Libraries, UNT Digital Library. https://digital.library.unt.edu
22. Gutierrez-Solana, F., & Elices, M. (1982). High-pressure hydrogen behavior of a pipeline steel. In C. G. Interrante & G. M. Pressouyre (Eds.), *Current solutions to hydrogen problems in steels* (pp. 181-185). American Society for Metals.
23. Álvarez, G., et al. (2019). Hydrogen embrittlement of structural steels: Effect of the displacement rate on the fracture toughness of high-pressure hydrogen pre-charged samples. *International Journal of Hydrogen Energy, 44*(29), 15634-15643.
24. Álvarez, G., et al. (2021). Hydrogen embrittlement testing procedure for the analysis of structural steels with small punch tests using notched specimens. *Engineering Fracture Mechanics, 253*, 107906.
25. Cialone, H. J., & Holbrook, J. H. (1988). Sensitivity of steels to degradation in gaseous hydrogen.
26. Zawierucha, R., & X. K. (2005). Hydrogen pipeline steels. *Materials Science and Technology, 3*(8), 79-90.
27. Duncan, A., Lam, P.-S., & Adams, T. (2007). Tensile testing of carbon steel in high-pressure hydrogen. In *ASME 2007 Pressure Vessels and Piping Conference*.
28. Oesterlin, H., Fischer, C., & Michler, T. (2024). Evaluation of lifetime predictions for future hydrogen pipelines by different fracture mechanics-based design codes. *Energy Technology, 2301012*. https://doi.org/10.1002/ente.202301012
29. Fischer, C., Fliegener, S., Oesterlin, H., Michler, T., Höhler, S., Mondry, A., & Ertault de la Bretonniere, P. (2023). Codes and standards for the fatigue-based design of hydrogen infrastructure components. *International Journal of Fatigue, 171*, 107564. https://doi.org/10.1016/j.ijfatigue.2023.107564.