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Sub-surface corrosion research on rock bolt system, perforated SS sheets and steel sets for the Yucca Mountain Repository — Quarterly technical report No. 1

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July 1, 2004 to September 30, 2004

Task No: ORD-FY04-019 and Document No: SIP-UNR-040

Prepared for U.S. DOE/UCCSN Cooperative Agreement Number DE-FC28-04RW12232

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October 7, 2004

**Task ORD-FY04-019 "Sub-surface Corrosion Research on Rock Bolt System, Perforated
SS Sheets and Steel Sets for the Yucca Mountain Repository"
(A Quality-Affecting Task)
Principal Investigator, Dhanesh Chandra
DOE Technical Representative, Jaime Gonzalez**
Quarterly Technical Progress Report
07/01/04 - 09/30/04

Statement of Work

The principal purpose of the cooperative agreement is to develop and continue providing the public and the Office of Repository Development (ORD) of the U.S. Department of Energy's (DOE) Office of Civilian Radioactive Waste Management (OCRWM) with an independently derived, unbiased body of scientific and engineering data concerning the study of Yucca Mountain as a potential high-level radioactive waste repository. Under this agreement, the University and Community College System of Nevada (UCCSN) will perform scientific or engineering research, and maintain and foster collaborative working relationships between government and academic researchers. The following describes the objectives of Task 019 "Sub-surface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets for the Yucca Mountain Repository" under the cooperative agreement. The objective of this task is to conduct corrosion research and predict the durability of rock-bolts and other underground metallic roof supports. Corrosion resistance of rock bolts (such as Swellex, CT, and hollow core anchor) made of high-strength low alloy steel, medium carbon steel (from the YM site), stainless steel, and other higher strength materials will be evaluated. In addition, investigators will examine underground tunnel roof support materials such as perforated stainless steel sheets, I Beams, and associated materials. The proposed research for this project is divided into eight different subtasks given as follows:

Subtask 1: Selection of New High Strength Steels, Stainless Steels for Rock Bolts, Steel
   Sets and Perforated Roof Supports
Subtask 2: Electrochemical Corrosion Tests
Subtask 3: Electrochemical Impedance Spectroscopy (EIS)
Subtask 4: Environmentally Assisted Corrosion Tests
Subtask 5: Hydrogen Permeation Studies
Subtask 6: Immersion Corrosion Tests
Subtask 7: Dry Oxidation Tests ASTM G-54
Subtask 8: Microstructure and Phase Characterization Studies
   This task is an essential part of the program to enhance the collaborative ongoing
   research between the UCCSN and the YMSCO during the five-year period of the
   cooperative agreement.

General Statements

- Technical Meeting and Presentation of the proposed research for this project to DOE-Las Vegas underground repository team for nuclear storage.
- Quality Assurance Training performed for all the staff working on the Yucca Mountain project
- Prepared and sent an SIP to Harry Read Center for QA approval - in final stages.
- Worked on two publications from the previous YM project research that are accepted and are in print. Both papers are peer reviewed. The paper in *Corrosion Science* (vol. 46

(2004) 2629-2649, Elsevier) is already printed already. The other paper is accepted and in Print in *Corrosion Journal*.

- Two other papers have been present at National and International Meetings.
- A Ph.D student (Mr. Josh Lamb) who worked on our previous project is actively working on our new YM project. Three new Ph.D graduate students have been recruited and are working on the project.

Progress for the Period 07/01/04-09/30/04:

- SIP was written and revised for reviews at HRC and DOE. The SIP is enclosed as an Appendix to the Technical Report. The SIP is in its final stages of approval.
- Worked on two manuscripts for publication in Corrosion and Corrosion Science.
- Presented (oral) paper at the Scientific meetings at the TMS International Meeting in New Orleans, Louisiana (September 26-29, 2004), and another one at the XIII International Meeting on Materials Research Congress (22-26 August 2004) in Cancun Mexico.

We have prepared the SIP which is enclosed in the Appendix. This SIP is in final stages of approval after revisions proposed by various reviewers. The QA research cannot start until HRC-DOE gives permission to UNR. At this time we are performing literature search, obtained samples of the rock bolts from Yucca Mountain sites of Swellex and Split sets.

APPENDIX

***Sub-surface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets
for the Yucca Mountain Repository***

Long-Term Drift Stability, Subtask: Corrosion Research on Rock Bolts

Scientific Investigation Plan for the YM project
-submitted to-
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United States Department of Energy-Merit Review Board
Through
Harry Reid Center For Environmental Studies- UNLV
4505Maryland Parkway
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Table of Contents

I. Introduction

The objective of the proposed investigation is to conduct corrosion research and predict the durability of rock-bolts and other underground metallic roof supports. In critical areas, it is possible to use highly corrosion resistant steels for rock bolts at Yucca Mountain (YM) repository, as the steel rock bolts, as well as other materials such as Bernold type shields for tunnels at YM site [1-4]. In addition, there is propensity for stress corrosion cracking as well as hydrogen induced cracking in rock bolts likely to occur in YM repository emplacement under drift conditions. As corrosion of these materials for underground support structures depends on the YM environmental constraints, loading, and temperatures, it is necessary to evaluate different materials with a wide range of corrosion rates for the rock bolts, particularly important for SCC and HE resistance which incorporate these conditions. Thus these studies will enable DOE to more accurately model for long-term predictions of drift stability during YM repository service. It is proposed that the following tasks be performed to further this goal. The tables below describe the proposed materials for the rock bolts and steel sets as well as the types of tests important corrosion tests of significance to determine the optimal materials for use at the YM underground repository.

We will start corrosion research using different types of tests on the proposed new alloys, Swellex, Split set, CT rock bolts, and rock bolts in service at YM test site for 5 to 7 year supplied by DOE. We will have a database for corrosion rates of materials important for repository as the work progresses. In the previous project we have studied medium carbon steel rock bolts, but there are still two important tests that we could not complete earlier in Task 18 due difficulties with machining and time constraints; these are *stress corrosion cracking* tests. Other test that remains is the *dry oxidation test*. Steels sets have been extensively studied in the Task 18 (2000-2003). Please see Figure1 RB and I-Beam). The proposed research for this project is divided into *eight different tasks* and summary of the tasks baseline YMP conditions, anticipated accomplishments. A body of evidence of corrosion rates
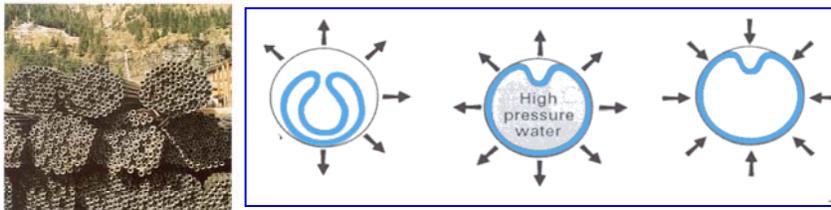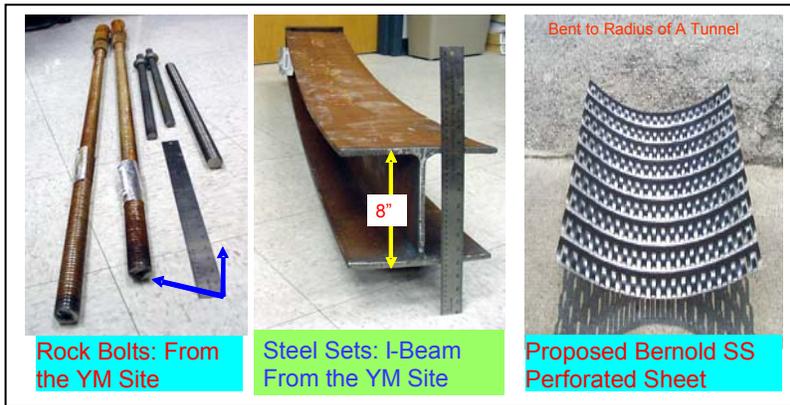
Figure 1. Photographs of rock bolts (left) and steel set (I-beam-middle) already being studied at UNR; we have a few more tests left on these materials to perform. The new materials are stainless and other highly corrosion resistant alloys for rock bolts, and steel sets (not shown here). We will also study new "Bernold type" perforated underground support steel sets (right). Swellex bolts stored at Mitholz on Alp Transit Lotschberg in Switzerland and expansion of Swellex bolt into contact with rock by high pressure water. (Bottom)

for various materials will be gathered and compared to a bench mark test. The value of each task is also indicated in this summary Table 1. Experimental procedures are listed in the later sections.
MCB = Medium Carbon Steel (Figure 1-left), LCS = Low Carbon Steel LCS (Figure 1-middle), BPRS= Bernold Perforated Stainless Steel Roof Support (Figure1-right), SS = Stainless Steel (not shown).

II. Scope, Objectives and Subtasks

II.1: Scope
Task: Sub-surface corrosion research on rock bolt system, perforated SS sheets and steel sets for the Yucca Mountain repository.

This work is subject to UCCSN QA requirements.

This work will collect corrosion results using both electrochemical and classical techniques.

This SIP presents an independent confirmatory study supporting previously gathered info. This will build upon previous work detailed in SIP-UNR-018 [1] and TR-03-017 [2]. This previous work was subject to UCCSN QA requirements.

II.2: Objectives
The following table details the objectives of the task.
Table 1: Summary of Subtasks

Subtask No. and Description	Types of Materials or Test to be performed	Anticipated Accomplishments
1. Selection of Alloys for Rock bolts, Bernold Type supports. (Also tests involving rock bolts already installed at YM test site for 5-7 years, if provided by DOE)	Rock Bolts (Swellex bolts, CT, and others supplied by DOE in addition to materials procured from vendors). Bernold type support material that has localized strain will be tested by using cold rolled steels (with different degree of hardness) commonly used for these supports. Cut-out samples from Bernold sheets will also be tested. (Screening test samples will be prepared on many samples either cut from new alloys, from actual rock bolts (new or on service at YM site), and Bernold type shields for comparison with baseline material.	Sample preparation for commercial rock bolts (RB) and potential new alloys for YM project to be tested in Task 2 and 6 for screening.
2. Electrochemical tests to evaluate corrosion rate and possible corrosion mechanisms	1. Polarization Resistance Tests [For corrosion rates (CR)] (a) Effect of Electrolyte, (b) Effect of Temperature, (c) Effect of Different Alloy material, (d) effect of work hardening. 2. Cyclic Polarization tests (limited tests) 3. Corrosion Potential vs. Time, *Constant Potential tests to obtain samples for XPS work for determination of corrosion mechanisms. Detailed tests will be made on samples that show promise with the lowest corrosion rates as compared to the baseline sample.	Determination of corrosion rates of different materials for YM site, to determine effect of temperature, concentration of YM waters, samples for mechanistic studies.
3. Electrochemical Impedance Spectroscopy (EIS)	These are complementary electrochemical tests to check for consistencies on underlined samples.	Checks the various effects obtained in Task No. 2 tests
4. Environmental Assisted Corrosion Tests (Combined electrochemical-mechanical tests under dynamic stresses)	Stress Corrosion Cracking and Hydrogen Embrittlement Studies will be preformed at UNR using a Slow Strain Rate Testing (SSRT) mechanical testing machine. New Nano mechanical tests will be performed to mechanically characterize thin corrosion films in-situ electrochemical test. In-situ applied potential tests (or open circuit), as well as ex-situ strain rate sensitivity tests will be performed on selected samples after the CR rates have been determined in Task No. 2.	This will give closest to near field test of mechanical behavior under the influence of the electrochemical environment experienced using YM waters for rock bolts.
5. Hydrogen Permeation tests	Devanathan-Staruchi Cell systems available at UNR will be used for this test on rock bolts and Bernold type sheet materials. A modified quartz cell will be used for (20-96°C) temperature tests. Important for hydrogen related cracking resulting failures of bolts under stress.	Amount of hydrogen trapped and transported in a metal.
6. Classical ASTM	YM Water Spray test or Fog Test with 100x YM water (using new	Corrosion rates by

Type Immersion tests	water chemistry to be furnished by DOE), Cyclic Humidity tests (G-60) classical tests to determine corrosion rates.	conventional ASTM tests using YM waters
7. "Dry" Oxidation tests	"Dry" Oxidation Tests will be performed on all alloys including LCS, MCS, 4340, and other RB supplied by DOE. More precise *Thermogravimetric Analyzer (TGA)* tests are designed to determine the weight changes in rock bolt* and other materials** as result of oxidation of materials (under controlled humidity). *Commercial Swellex, CT and other rock bolts from DOE and C-22** with lowest corrosion rate.	Dry oxidation tests will be performed using YM water vapor to test the amount of weight changes during exposure of rock bolts and other materials.
8. Characterization	Metallographic analyses of samples before and after corrosion tests using standard optical microscopy. Scanning Electron Microscopy (SEM) and energy dispersive analyses (EDS) will be used to evaluate chemical compositions at UNR. Transmission Electron Microscopy (TEM) at either UNR or Uni. of Illinois/ LBNL. Phase Analyses by X-ray diffraction at UNR at temperatures. X-ray Photoelectron Microscopy (XPS) at PNNL (Washington). Optional synchrotron- in-situ electrochemical tests (arrangements yet to be made) will be performed at Brookhaven National Laboratory. *These tests will be performed on as needed basis on selected samples.*	These will give mechanisms of corrosion using different test conditions to observe the effect of material and environment on corrosion rates.
Note No.1. As we gather the body of evidence, we will summarize the corrosion rates obtained from different rock bolts and other supporting materials being considered for the YM project, as well as new materials that will be investigated. The corrosion rates will be compared with benchmark "rock bolts" and sheet materials.		
Note No. 2. The approximate field conditions will be used by testing the metal samples at different temperatures ranging between room temperature to 96°C by adjusting the water chemistry furnished by DOE in 1x, 10x and 100x concentrations.		

II.3 Subtasks

All subtasks are subject to Q requirements. Any subtask that is changed to non-Q at any point during the task requires notification of the DOE Technical Task Representative with justification.

Subtask1. Selection of Alloys and Machining of Specimens for Rock Bolts, Steel Sets, Wire Meshes and other Perforated Sheets Roof Support Materials. (CR= Corrosion rate).

No.	Type of Material	Alloys	Screening Test Methodology	Selection of Materials/Sample machining for Corrosion tests
A.		Medium carbon steel Rock Bolts (RB) (*Relatively High CR-Baseline material*)	ASTM G-5 Electrochemical tests in 1x YM Water DTN No. LB0101DSTTHCR1.001	Samples to be machined for stress corrosion and dry oxidation tests
B.	Commercial Rock bolts to be obtained from DOE	Swellex Rock bolts, Super-Swellex and new Swellex Mn 24 type (From either YM site or will be procured)	or updated chemistry at 25, 45, 65, 85° and others as needed. (Please Note: After the screening tests are performed we will	Samples to be machined for Stress corrosion/hydrogen embrittlement, Potentiodynamic tests for estimating corrosion rates, Classical ASTM Immersion, cyclic humidity, and fog tests.
C.		Split set friction rock stabilizers from DOE	confer with DOE to proceed with the detailed	Sample machining for Task No.2.

D.		CT Rock bolts from DOE	tests such as long-term ASTM classical tests, and on selected materials.)	Sample machining for Task No. 2
		Faceplates for Swellex bolts		Samples for task No. 2 and 4
E.	Manufactured in Switzerland →	Bernold Type reinforcement steel lining. (Strain hardening effects expected in the punched-out areas will be evaluated)		Sample machining for Task No.2 and 4.
F.	Plates or rods will be procured for tests from vendors. Efforts will be made to obtain commercial RB's, if available, for tests.	High Strength Low Alloy 4340 Steel for RB		Samples for screening test in Task No. 2
G.		SS 316Lfor RB		Samples for screening tests
H.		Other Commercially (Ex:RB C-22 type available stainless steel Rock bolts and Sheets		Samples for screening tests
I.		Duplex Stainless Steels: AL6XN, 254-SMO for RB and BPRS		Plates and rod specimens to be machined for test in Task No. 2 and 4.
J.		C-22 for RB (*Low CR-Baseline Material*)		Plates and rod specimens to be machined for test in Task No. 2, 4 and 5.

Tasks	**Sub*task No. 2 and 3 Electrochemical Tests***
	Alloys listed in task No.1
	Standard Salt environment ASTM baseline tests will also performed as reference.
Task No. 2A.	Electrochemical tests involving, Corrosion Potential, E_{corr} vs. Time.
Task No. 2B.	3x Polarization Resistance Tests (ASTM G-59) *1. Effect of Electrolyte Concentration 2. Effect of Temperature 3. Effect of Different Alloys. 4. Effect of Work Hardening*
Task No. 2C.	Cyclic Polarization (ASTM G-61)
Task No. 2D	Constant Potential Tests and Samples will be examined by XPS for surface analyses depth profiles
Task No. 3	Electrochemical Impedance Spectroscopy (EIS) The passivation phenomenon in corrosion process. This is a complimentary test performed at different temperatures and YM water concentrations.

Task No. 4	***Subtask No. 4 Environmental Assisted Corrosion (EAC) Tests***
	Standard environment ASTM baseline tests will also performed as reference.
	The low carbon steel (already investigated) will used as a reference or baseline for these studies.
	Alloys Listed in Table 1.
Task No. 4A	Stress Corrosion Cracking and Hydrogen Embrittlement Studies will be preformed UNR using the SSRT mechanical testing machine.
Task No. 4B Task No. 4C	Perform Using Slow Strain Rate Testing (SSRT) In-situ applied potential tests (ASTM G129) Ex-situ Strain Rate Sensitivity Tests (Samples will be charged at 5 mA/cm^2 cathodically for one week in 5% H_2SO_4 and mounted on the mechanical testing machine to obtain stress strain curves in air.

Subtask No. 5 Hydrogen Permeation
YM rock bolt MCS, Low Alloy High Strength Steel (4340), 316L and Related Commercial SS Rock Bolts, and C-22

Task No. 5	Devanathan-Staruchi Cell systems available at UNR will be used for this test. We have the IP's for the use of this instrument and tests have already been performed on rock bolt and I-beam materials. A modified quartz cell will be used for (20-85°C) temperature tests.

Subtask No. 6. Immersion Corrosion Tests
YM rock bolt MCS, Low Alloy High Strength Steel (4340), 316L and Related Commercial SS Rock Bolts, RBC-22 SS RB, Duplex Stainless Steel, and C-22

Task No. 6A.	YM Water Spray test or Fog Test ASTM (No controlled Potential)
Task No. 6B.	ASTM G-31/G1 at 100x YM water (or New water chemistry to be furnished by DOE)
Task No. 6C.	Cyclic Humidity test (G-60)

Task No. 7	***Subtask No. 7 Dry Oxidation Tests***
	YM rock bolt MCS, Low Alloy High Strength Steel (4340), 316L and Related Commercial SS Rock Bolts, RBC-22 SS RB, Duplex Stainless Steel, and C-22
	Dry Oxidation Tests. In this we will perform standard ASTM G-54 for all alloys including LCS, MCS, 4340, and others. Based on the initial results of the G-54 tests, we may perform additional (and more precise) *Thermogravimetric Analyzer (TGA)* tests to determine the weight loss rock bolt MCS steel, I –beam steel, stainless steels, and C-22.

Task	**Subtask No. 8. Characterization**
	YM rock bolt MCS, Low Alloy High Strength Steel (4340), 316L and Related Commercial SS Rock Bolts, RBC-22 SS RB, Duplex Stainless Steel, and C-22
	Tests on as needed basis
Task No. 8A	Metallographic Analyses of the samples before and after corrosion tests using standard optical microscopes with digital photography at UNR.
Task No. 8B	Scanning Electron Microscopy (SEM) and energy dispersive analyses (EDS) will be used on as needed basis to evaluate corrosion products and chemical compositions at UNR. Transmission Electron Microscopy (TEM) at either UNR or Uni. of Illinois/ LBNL.
Task No. 8C	Phase Analyses by X-ray diffraction Analyses at UNR
Task No. 8D	X-ray Photoelectron Microscopy (XPS) at PNNL

III. Methods and Approach
III.1 Description of experimental subtasks

Subtask 1: Selection of Materials for Rock Bolts, Wire Meshes and other Roof Support Materials
It is proposed that corrosion resistant alloys will be examined as described in Table in Task No.1. It is expected that information from this electrochemical screening test will give us a general idea of the corrosion rates under different YM water concentrations. This will provide results on corrosion susceptibility of a variety of materials under varying conditions. The spectrum of alloys proposed will give us a prediction capability under different YM water concentration as well as the temperatures. This task involves work in Task No.2.

Subtask 2: Electrochemical Tests
It is proposed that electrochemical tests will be conducted at a computer controlled work station, consisting of a PC loaded with a Potentiostat and a software (Gamry Instruments, model DHC2, PC4(750)/DC105) at our UNR Corrosion laboratory. The experimental set-up for both, polarization and impedance spectroscopy, is illustrated in Figure 3 (taken from Dr. Yilmaz's UNR Dissertation, 2003 on YM project). The simulated waters in the test cell will be conditioned (aerated/de-aerated) by purging a proper gas (oxygen/nitrogen) for two hours, before specimens are immersed. Then freshly finished wet-ground specimens with the final 600-grit emery paper were immersed into the conditioned waters. From previous experience, they should reach stable open circuit potential (corrosion Potential or Ecorr) within two hours in the conditioned test solution. Then the polarization scanning is started. The potentiodynamic method (ASTM G59) has previously been found useful to obtain polarization resistance values. A platinum sheet is used as a counter electrode and solution bridge overcame the possible noise and instabilities mostly associated with this particular method with low solution concentration combined with high temperatures (65-85°C). Potentiodynamic scans should be started with 300 mV cathodic over potential, and raised from this value linearly up with a sweep rate of 0.166 mV/s. These scans can be extended through the pitting potential regions, until the currents reach a value of approximately 100mA/cm^2. The linear sections of the polarization curves (E vs. I), around 25 mV anodic and cathodic over potentials, will be selected to determine the polarization resistance (R_p) values. Corrosion rates are calculated using those R_p values determined by the Gamry software, out of manually selected linear portions of the polarization curves, 25 mV above and below the E_{corr} of the specimens. The rate calculations are carried out by the following equation [3,4]:

$$\text{Corrosion Rate   (CR)}$$
$$CR=3.27\times10^3.(i_{corr}.EW)/\rho \tag{1}$$

where, $i_{corr} = 10^6 . B/R_p$ and B is constant involving the Tafel constants.

Electrochemical Measurements for the Corrosion Testing
These studies will be performed to measure the corrosion rate in the aqueous solution. In this the sample is charged anodically to measure its anodic polarization curve and then the cathodic polarization curve by charging it cathodically. Tafel plots can be extrapolated from these polarization data. The intersection of the cathodic and anodic Tafel curve will give us the corrosion rate of the specimen. The hypothetical cathodic and anodic polarization curves are shown in Figure 2. The polarization resistance, R_p, of a corroding electrode is defined by the equation below as the slope of a potential versus current density at i= 0 [3,4].

$$R_p=(\delta\Delta E/\delta i)_{i=0, dE/dt\to0} \tag{2}$$

The current density is given by, i. The corrosion current density, i_{corr}, is related to the polarization resistance by the coefficient B (Eq.3).

$$i_{corr} = 10^6 B/R \tag{3}$$

where, B is given by Eq. 4

$$B = \beta_a \beta_c/[2.303(\beta_a + \beta_c)] \tag{4}$$

where, β_a β_c are anodic and cathodic Tafel constants respectively. The corrosion rate (CR) can be determined by Eq.5:

$$CR = 3.27 \times 10^{-3}\ i_{corr}\ EW/\rho \tag{5}$$

Experimental Procedure

A schematic of typical polarization diagram is shown in Figure 2, and the circuitry for the polarization measurements on a specimen or working electrode (WE) in Figure 3. A filtered direct current (DC) power supply, PS, supplies current (I) to the working electrode, through a second counter or auxiliary electrode (AUX). The potential of the working electrode is measured with respect to reference electrode with a series-connected potentiometer (P) and electrometer null detector (N). The test cell will be constructed to include the test electrode, two auxiliary electrodes, and a luggin capillary with salt-bridge connection to the reference electrode, inlet and outlet for an inert gas, and a thermometer. The suitable test cell that has been constructed at UNR is shown in Figure 4. A 1-liter round bottom flask has been modified by the addition of various necks to permit the introduction
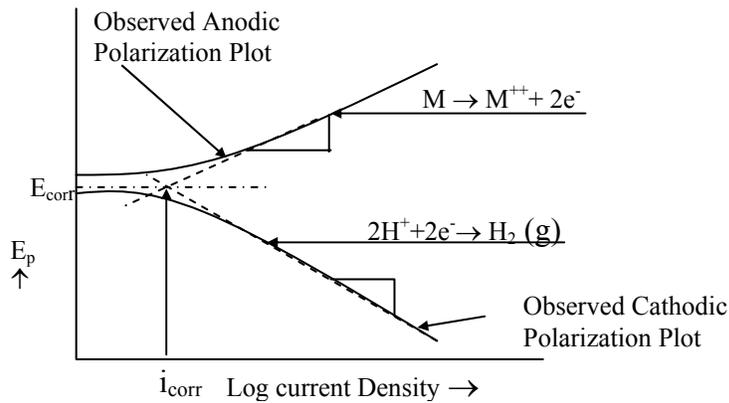

Figure 2. Hypothetical Cathodic and Anodic Polarization Diagram.

of electrodes, gas inlet and outlet tubes, and a thermometer. The Luggin probe-salt bridge separates the bulk solution from the saturated calomel electrode, and the probe tip can be easily adjusted to bring it in close proximity with the working electrode.
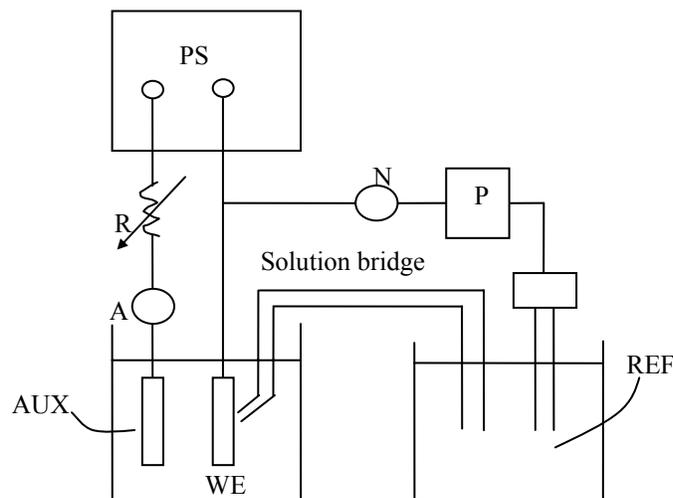

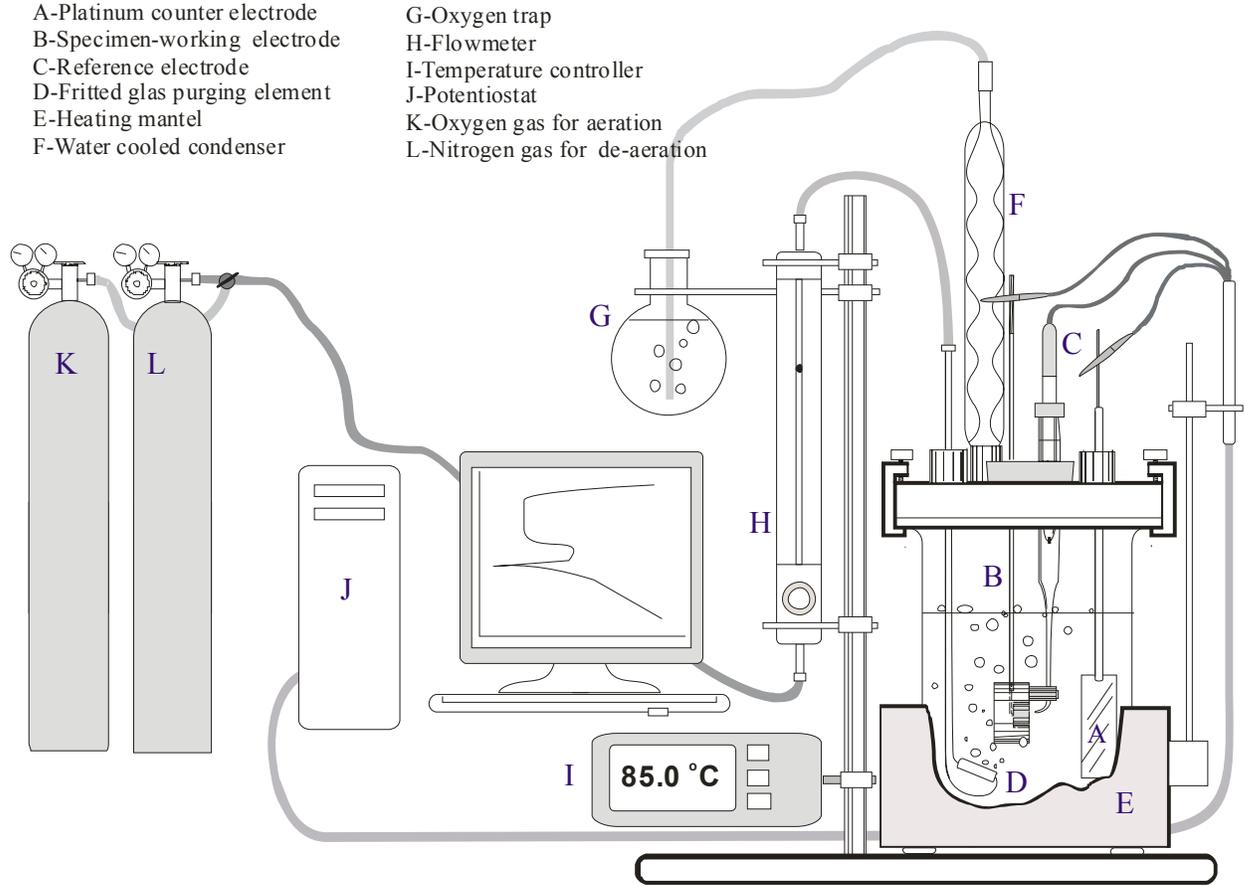Figure 3. Schematic of circuitry for polarization measurements.

A-Platinum counter electrode
B-Specimen-working electrode
C-Reference electrode
D-Fritted glas purging element
E-Heating mantel
F-Water cooled condenser

G-Oxygen trap
H-Flowmeter
I-Temperature controller
J-Potentiostat
K-Oxygen gas for aeration
L-Nitrogen gas for de-aeration

Figure 4. Experimental set-up for potentiodynamic linear polarization and impedance spectroscopy.

Task 3. Electrochemical Impedance Spectroscopy

It is proposed that electrochemical impedance spectroscopy (EIS) be performed at the corrosion potentials (open circuit potentials) of the test specimens using the same cell set-up and the same software as used in the polarization studies, which is introduced in Figure 4. Polarization resistance values can be determined from the real axis intercepts of the obtained semicircles of Nyquist plots of the impedance scans. Please see Figure 5 for polarization curve and the point A through E and the corresponding Nyquist plots in Figure 6. These tests will be used to determine the electrochemical interface leading to activation and passivation events on the carbon and other steel surfaces during polarization experiments. In general, impedance $Z(\omega)$, may be expressed in terms of real, $Z'(\omega)$ and imaginary $Z''(\omega)$, components [3,4].

$$Z(\omega) = Z'(\omega) + Z''(\omega) \tag{6}$$

The Stern-Geary equation (Eq. 6) provides a direct relationship between the steady state corrosion current and the resistance across the interface, which can be expressed as:

$$i_{corr} = \left[\frac{\beta_a \beta_c}{2.303(\beta_a + \beta_c)} \right]\left(\frac{1}{R_p} \right) \tag{7}$$

where, β_a and β_c are the Tafel constants for the anodic and cathodic partial reactions, respectively and R_p is the

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polarization resistance. Polarization resistance is given by the difference of the measured impedance at sufficiently low and high frequencies:

$$R_p = |Z(j\omega)|_{\omega \to 0} - |Z(j\omega)|_{\omega \to \infty} \qquad (8)$$

At University of Nevada, Reno we have Gamry Potentiostats with which we can perform Impedance spectroscopy analysis ASTM G106. Nyquist and Bode plots will be obtained for rock bolt steels in YM environment at different concentrations of ionic salts (1x to 500x). To understand the effect of applied potentials on the passivation tendency, and its influence on the impedance change, experiments will also be conducted at different potentials by fixing the electrode potential potentiostatically. Influence of temperature will also be studied (45, 65 and 85°C). Representative plots obtained in our laboratory showing the effect of impedance changes at different potentials applied on the carbon steel electrode exposed to 3.5% NaCl solution with different anions at pH 8 is displayed in Figures 5 and 6.
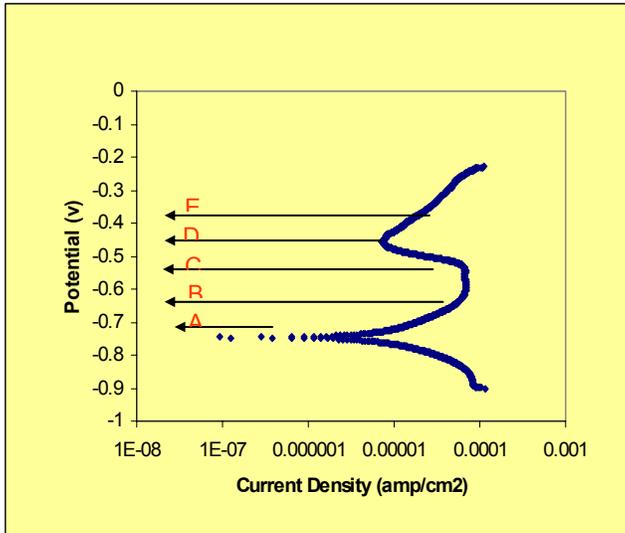

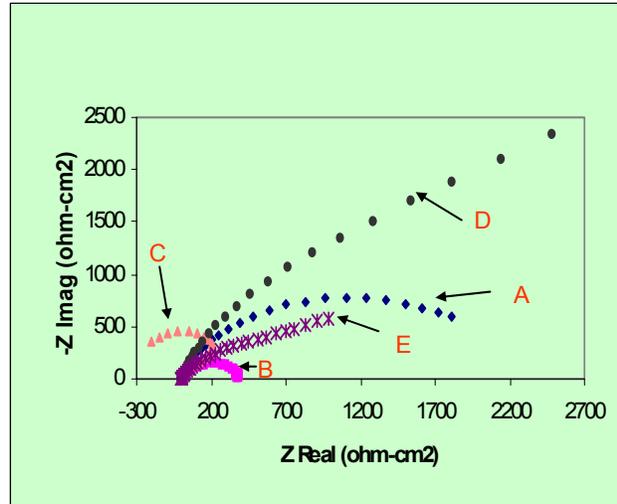Figure 5. Polarization behavior of carbon steel in 3.5% NaCl + silicate + bicarbonate+ sulfate, pH = 8

Figure 6. EIS performed at potentials corresponding to points marked in Figure 5.

Subtask 4: Environmentally Assisted Corrosion Tests

A. Stress-Corrosion Cracking and Hydrogen Embrittlement Tests: It is proposed that the University of Nevada, Reno's screw–driven SSRT universal machine (United STM-10ES) along its software and a Gamry Potentiostat (DC-105) be used for environmental cracking susceptibility determinations for I-beams. A schematic of the experimental set up is given in Figures 7 and 8. Also, details of the experimental cell and the set-up are given in Figure 8. All SSRT of I-beam (low carbon steel) were carried out with 100X simulated YM water at two different temperatures such as 25 °C and 85 °C; these will be followed for the rock bolts, studies yet to be performed. The simulated waters will be conditioned (de-aerated) by purging nitrogen gas for at least one hour before specimen immersion. Then, freshly prepared wet-ground specimens with a 600-grit emery paper finish were immediately installed into the cell through the top and bottom central necks using o-ring compression fittings. Then the conditioned de-aerated 100X YM water transferred to the test cell within 10 to 15 seconds without exposing it to air for too long. The cell openings are then closed quickly by threaded o-ring fittings and neck joints, which hold the entire elements of the cell sealed. The installation of the cell on to the universal joints of the SSRT machine should not exceed a total of 4-5 minutes after finishing the specimen preparation. By this rapid installation, possible de-aeration loss of the conditioned test solution, subject to effect the open circuit potential of the LCS by letting excessive oxygen in to the cell, should be prevented. After setting the temperature controller to the desired test temperature, open circuit potential monitoring with the Gamry Potentiostat is started. From previous experience, the open circuit potential (E_{corr}) will stabilize around -700 mV$_{Ag/AgCl}$ while the temperature will have already reached to the desired values to an accuracy of ±0.1 °C. De-aeration by purging nitrogen gas into the test solutions was continuous throughout the tests at a flow rate of 150 ml/min. Then, the conditioned specimens in the test solutions

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were charged for approximately 45 minutes by imposing desired potentials through Potentiostatic method, until the current values were stabilized prior to monitoring stress-strain curves and currents.

Then the SSRT is started with appropriate strain rates from 10^{-3}/s to 10^{-7}/s. During the tests, the changes over the test solutions and specimens were examined, and recorded. After the failure of the specimens, the cell was uninstalled and the pH changes of the test solutions were recorded. Specimens were washed with distilled de-ionized water, and acetone and alcohol, and retained to carry out scanning electron microcopy (SEM), energy dispersive spectral (EDS) analysis, optical microscopy, and reduction of area measurements.

B. _New Nanotechnology Mechanical Testing machine for Corrosion testing_: We are proposing to buy a new MTS XP machine to perform nanoscale mechanical tests. These are particularly useful for corrosion films that are formed in different electrolyte conditions by characterizing surfaces in a few nanometers, such as, for corrosion product formed (dynamically) for properties like hardness and modulus of elasticity can affect the performance of materials under loads. (We would still like to do these tests, as they will be useful for smaller parts. This was expected to be purchased but there were significant budget cuts in the first year, so we cannot buy this machine. Making an indentation with a controlled force, with high displacement and load resolution, while continuously monitoring the displacement of the indenter (the depth of the indentation), produces data from which the Young's modulus, fracture behavior, hardness of the material and other mechanical properties may be calculated. This capability allows for the measurement of elastic properties of thin films as well as plastic properties, according to the manufacturers. These machines are in use at several Universities and very good data is being produced in solution environments. The new capabilities of the nano mechanical system will be explored simultaneously with the other proposed experiments
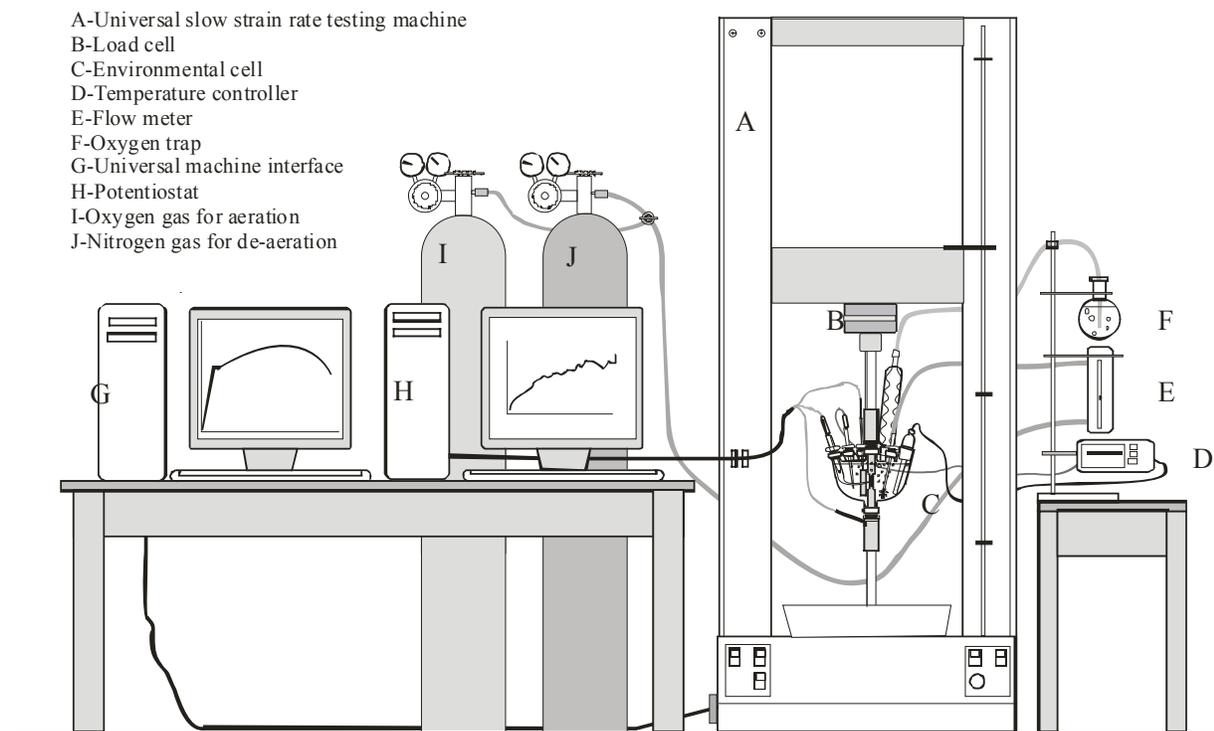

Figure 7.Experimental set-up for environmental cracking, such as SCC and HIC, and strain aging determination [5].

A-Cylindirical specimen-working electrode(WE)

B-Platinum counter electrode

C-Ag/AgCl reference electrode

D-Luggin probe

E-Quartz immersion heater

F-Fritted end purging capillary

G-Electrolyte

H-Water cooled condenser

I-Universal joints

J-O-ring seal threaded joints

K-Thermowell

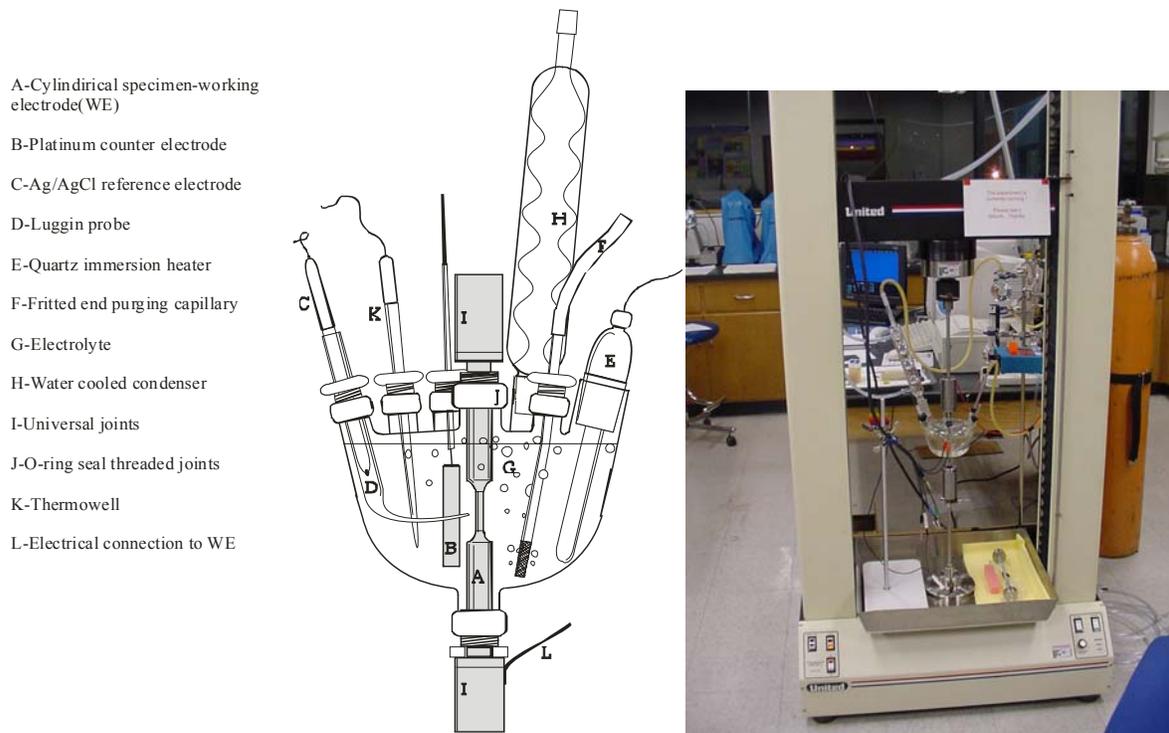L-Electrical connection to WE

Figure 8. A schematic of the SSRT test cell with its components (left) and the SSRT machine (right) equipped with the environmental cracking cell during an ongoing test.

(Please see equipment justification section), listed below with YM electrolyte, where needed. A photo of this equipment with environmental chamber is shown in Figure 9.   The table below lists the main features of the instruments besides standard software.
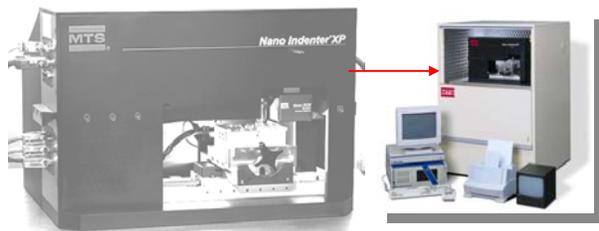
Figure 9. Nano MTS machine showing the basic instrument and the enclosure. (Courtesy: MTS Corporation.)

Continuous Stiffness Measurement (CSM)	This micro mechanical CSM technique allows measurement of stiffness continuously during the indentation, i.e., at every point that a load-displacement data pair is taken, and the stiffness is directly measured. In this CSM method an oscillating force is imposed on the indenter while monitoring its response displacement. This displacement response is sinusoidal in nature due to the nature of sinusoidal excitation force, and is out of phase by some amount. The phase and amplitude of this response are characteristic of the material's properties as it is a direct measure of the stiffness of contact and the damping of the material at the frequency of the oscillation.
Dynamic Contact Module (DCM)	For thin films, such as corrosion products, we can use this nano-indentation head available is part of XP system for very low load situations such as thin forms formed during corrosion processes. The Nano DCM at right is mounted in a Nano Indenter XP system as an additional indentation head to the standard indentation the Nano DCM head can be accessed for testing as easily as the standard head, and multiple samples as well as multiple indentations on each sample to gives statistically significant data required in Nanoindentation. (Option)
Lateral Force Measurement (LFM)	Add the capability of scratch and friction testing, surface profiling and wear testing to make the system n added tribological tool.
High Load	The high load head takes the load-controlled depth-sensing indentation testing to over a

Indentation	kilogram for indentation cracking (fracture toughness) and micro-hardness simulation without changing the indenter head.
Nano positioning Stage	The XP system has a high precision and accuracy in positioning, the positioning system to optimize positioning performance.
Temperature Humidity Chamber	Humidity is controlled between 20 to 100% and Temperatures 10 to 100°C.

Subtask 5. Hydrogen Permeation in Alloys

Hydrogen permeation experiments were performed as per ASTM G148 in a system with two compartments separated by a bipolar membrane made of carbon steel is shown in Figure 10 and 11. Hydrogen permeation samples of 1.5 mm thickness were fabricated from rock bolt carbon steel rods and also from steel sets. These samples, coated with Ni plating on the exit side by electrolytic plating, are membrane was polarized as the anode (oxidation cell). The electrolyte on the charging or entry side and the oxidation or exit side of the cell was deaerated in 0.1M NaOH solution by using nitrogen gas prior to the experiments and during the permeation period on both the compartments. Hydrogen charging was done by galvano-static charging at an applied cathodic current of $1mA/cm^2$ and the anodic side of the specimen was fixed at a potential of + 0.28 V with respect to Silver-Silver Chloride (Ag-AgCl) to enable the oxidation of atomic hydrogen that diffused through the membrane. The anodic side of the membrane was electroplated with Ni to retard the corrosion reaction and to obtain steady state anodic background current at that potential. To enhance the hydrogen absorption, 30mg of arsenic trioxide (AS_2O_3) was added in the charging cell as a hydrogen recombination poison. The effect of trapping was investigated by recording the hydrogen permeation transients repeatedly on the same specimen after attaining the steady state current. In some experiments, to understand the effect of simulated YM repository water concentrations on the hydrogen permeation, experiments were done by changing the charging environment to simulated YM ground water instead of 0.1M NaOH. The electrolyte concentration was varied by increasing the dissolved ionic concentrations ranging from 1x, to 100x.

To calculate the critical hydrogen concentration, a third permeation can be obtained after completing the second transient at an applied current density of $5mA/cm^2$ using 1N H_2SO_4 solution. The calculation of C_k is performed according to the method proposed by Pressouyre et al. [5]. The depth of the crack is measured after completing the third transient by diametrically cutting the specimen followed by metallographic polishing and etching.

Data Analysis
The hydrogen permeation rate $(J_\infty L)$ (mol H cm^{-1} s^{-1}), effective diffusivity (D_{off}) (cm^2 s^{-1}) and solubility (C_o) (ppm) is calculated using the following relations

$$J_\infty L = i_\infty L/nF \qquad (12)$$

Where J_∞ is the steady-state flux, L is the specimen thickness in cm, i_∞ is the steady-state permeation current density in A/cm^2, "n" is the number of electrons transferred, F is the Faraday's constant (96485 Coulombs/mol.)

$$D_{eff} = L^2/6t_L \qquad (13)$$

Where t_L is the lag time $(0.63J_\infty)$

$$C = J_\infty L/D_{eff} \qquad (14)$$

The critical hydrogen concentration is the sum of hydrogen with in the steel at a depth x and is given as

$$C_k = C_i (x) + C_r (x) + C_d(x) \qquad (15)$$

Where, $C_{i,r,d}(x)$ = irreversible, reversible and diffusible hydrogen concentration. The difference in hydrogen concentration measured from the first and second transient would yield irreversibly trapped hydrogen (C_i). According to McNabb and Foster [6], the amount of irreversibly trapped hydrogen is independent of C_d and x. However, reversibly trapped hydrogen is considered to be in equilibrium with diffusible hydrogen and is given as follows:

$$C_r(x) = K C_d (x) \qquad (16)$$

Where C_d is the average diffusible hydrogen concentration measured from the degassing curves of the first and second transients. The difference between hydrogen concentration measured from second transient and the average
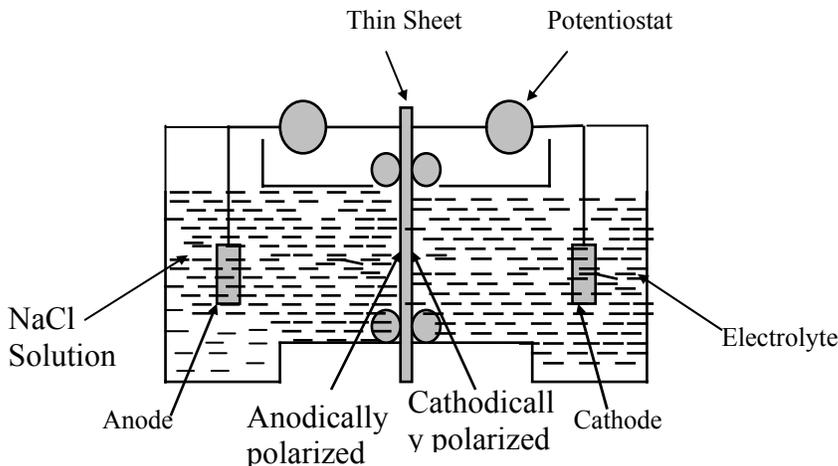
Figure 10. Devnathan-Staruchi cell schematic (left). Figure 11 Photograph of the actual hydrogen permeation cell [ASTM G148].

diffusible (C_d) content can provide the reversible (C_r) hydrogen concentration.   The constant K can be obtained from C_r/C_d. Now the diffusible hydrogen concentration at a depth x can be obtained as (Eq.14):

$$C_d(x) = C_o(1-x/L) \qquad (17)$$

Where, x is the depth of the crack, L is the thickness of the membrane and C_o is the diffusible hydrogen concentration obtained from the flux values measured for the cracking curve. The total amount of hydrogen at depth "x" where crack was found is given in the equation (Eq. 15):

$$C_k(x) = C_o(1-x/L)(1+K) + C_i \qquad (18)$$

Subtask 6. Immersion Corrosion Tests

Simple lab immersion and YM water spray (fog) specimens were machined out of the I-beam and rock bolt in the form of coupons and strips of 0.5 mm thickness and various surface areas, as shown in Figure 12. The specimens for cyclic humidity are shown on the right. The specimen preparation method met the standard practices ASTM G31 and G85, for simple lab immersion tests, and will be used for the new alloys.
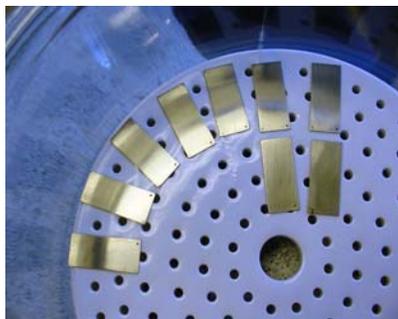
Figure 12. Coupon specimens for laboratory immersion and salt spray tests for rock bolts and I-beams. Similar specimens have to be prepared for the new specimens proposed in this study.

Subtask 6A. Salt (YM Water) Spray (Fog) Test [ASTM G-85]

These studies will be performed in order to determine the corrosion rate and resistance of the steel sets and rock bolts to the water vapor phase in the Yucca Mountain Repository. The corrosion rate in these studies will be calculated by the weight loss measurements as explained in the above Tasks. In this the steel coupon is exposed to the atomized spray of water (Figure 13 and 14). The water that is being atomized and sprayed will be of the same composition as that in the Yucca Mountain Repository. The apparatus of a salt spray (Fog) testing will consists of a

Fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control. The design of the chamber is so made that drops of solution that accumulate on the ceiling or cover of the chamber do not fall on the specimens being tested. And also the drops from the coupon are protected from falling down again to solution reservoir for re-spraying. The compressed air supply to the nozzles for atomizing the salt solution will be free of oil and dirt and maintain the air supply between 69 and 172 kPa. Temperature of the chamber will be about 100°C. Two clean Fog collectors will be placed within the exposure zone so that no drops of the solution from the test specimens or any other source can be collected. The sample taken out from the chamber are cleaned in accordance to the ASTM G-1 and weighed and measured for the corrosion rate of steel coupon in the fog of water.

Figure 13 (top left). Exposure chamber and solution reservoir used for salt spray tests.

Figure 14 (middle and right). Salt spray coupon specimens before Middle photo) and after (right photo) these tests were carried out at 35 $^\circ$C with 1X YM water spray at National Exposure testing, Sylvania, Ohio.

Subtask 6B. Aqueous Immersion Testing (ASTM G-31)

These studies will be performed for the aqueous solution in the Yucca Mountain Repository. These studies will be conducted by immersing the steel coupons in the aqueous environments (water) of the Yucca mountain repository. This method is also mass loss method as explained in the above Tasks. Coupon corrosion testing is predominantly designed to investigate general corrosion.

The apparatus used for the laboratory immersion testing is as shown in the Figure 15, which consists of kettle or flask of suitable size, a reflux condenser with atmospheric seal, valve for controlling the aeration, temperature-regulating device, heating device, and specimen support system. Strip coupons 50x.25x.1.6 or 3 mm may be preferred as corrosion specimens. Three such coupons will be employed with one completely immersed and other partially immersed and third one totally out of solution.

This steel coupon will be exposed to the Yucca mountain repository composition. Temperature of the water will be maintained around 80°C. Slight aeration will be provided to get similar conditions for those in Yucca Mountain. But specimen will not be located in the direct air stream from the spacer. The exposure of the specimen varies from 2-7 days. The sample will then be taken out and cleaned as explained in ASTM-G1. The corrosion rate (CR) will be measured by the weight loss measurement according to ASTM G-31:

$$CR = (K .x. W)/(A .x. T .x. D) \qquad (19)$$

where, K = a constant, T = Time of exposure in hours to the nearest 0.01hour, A = Area (cm^2), W = Mass loss in grams, to nearest 1 mg, D = density (g/cm^3).

A-Non-immersed specimen
B-partially immersed specimen
C-Fully immersed specimen
D-fritted glass purging element
E-Plug specimen holder
F-Thermowell
G-Water cooled condenser
H-Flowmeter
I-Oxygen trap
J-Oxygen gas for aeration
K-Nitrogen gas for de-aeration
L-Temperature controller
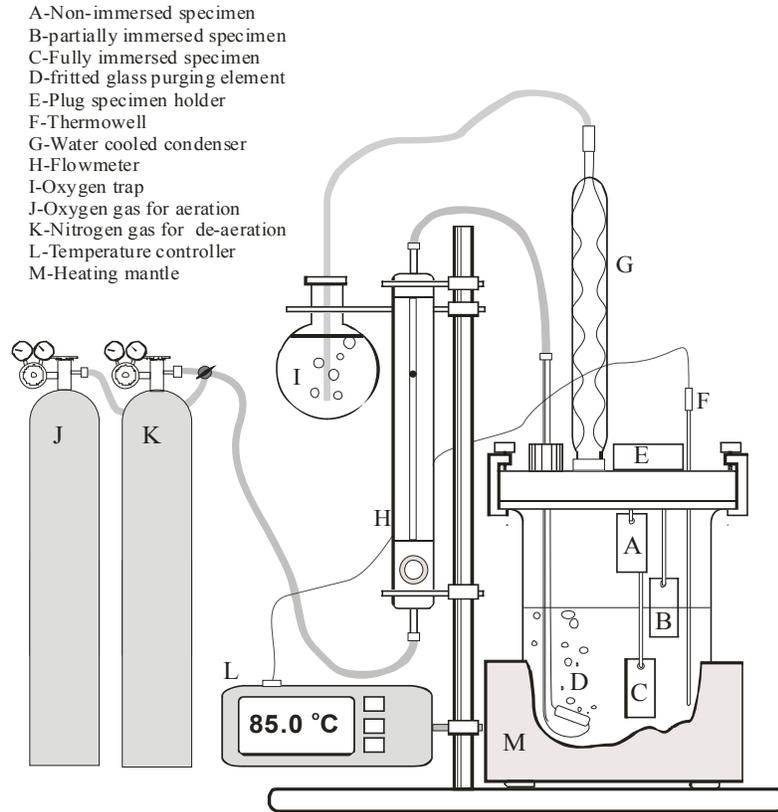M-Heating mantle

Figure 15. Experimental set-up for lab immersion corrosion testing in a sealed glass reaction flask (from Dr. Yilmaz Ph.D. dissertation at UNR, Prof. Chandra's YM group).

Hygrometer

Specimen

Humidifying Tower

Leveling Bottle of Dip Solution at 52 ^{o}C
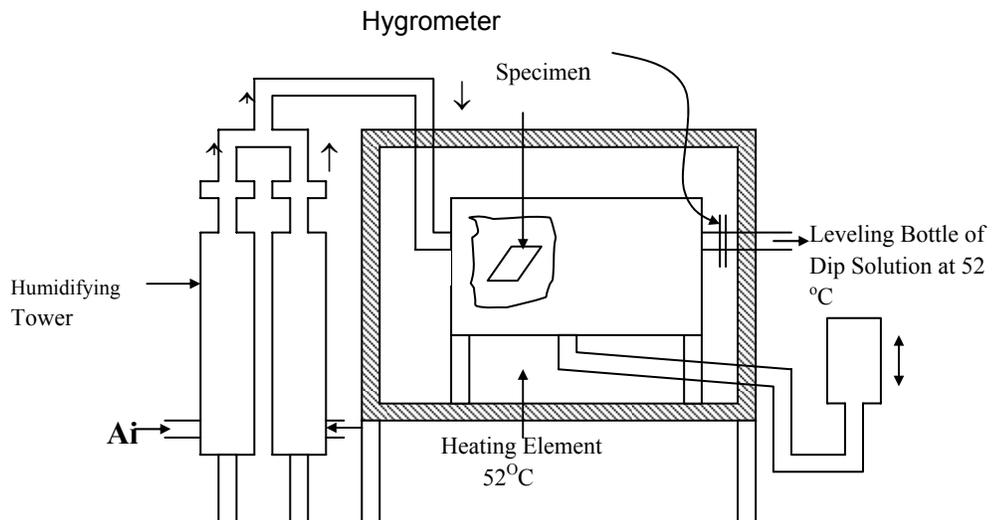
Ai

Heating Element 52°C

Figure 17. Schematic of humidity cycle profile for these experiments in Figure 29.
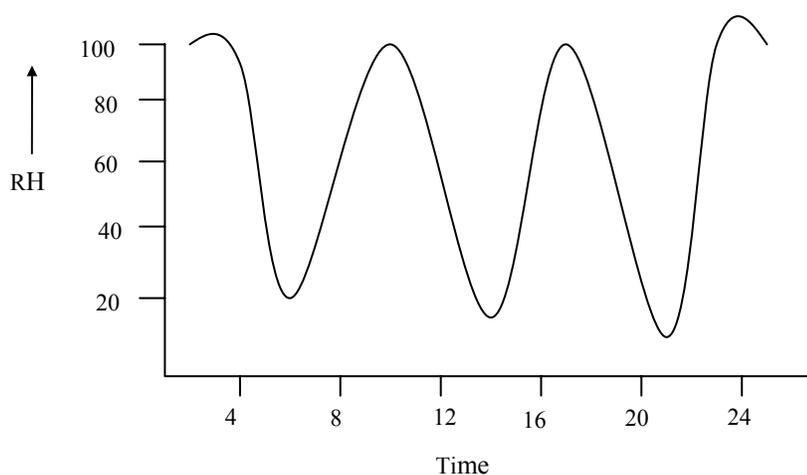
Figure 16. Schematic of Humidity chamber for new alloy testing [From ASTM G-60].

Subtask 6C: Cyclic Humidity Tests (ASTM G-60)

Cyclic humidity tests are proposed for the alloys in the present. A schematic of the apparatus is shown in Figure 16. This apparatus is available for us at MATCO Corporation. and will be used for our work (please see the quotation for the use of this apparatus). In this study, humidity cycle of 8-hour, three times/day, as shown in Figure 17. The ASTM standard specifies 52°C as temperature for testing for a period of 100 days.   The cyclic variation of the humidity can be obtained by variation of the temperature of the water in the humidifying tower. The range of relative humidity can be extended by adding a drying period to the humidity cycle. The control circuit will be arranged such that the air will be switched to the drying train when the relative humidity has decreased to 50%. The drying train can be a desiccating tower containing anhydrous calcium sulfate. The relative humidity will be maintained in between 50-100% or more for each cycle. The corrosion rate will be measured as mentioned in the above studies. This test method includes procedures for conducting cyclic humidity tests.   Specimens will then be prepared, cleaned, and evaluated in accordance to ASTM-G1. Evaluation of the corrosion rate will be done by weight loss measurement (penetration per unit time, mm/yr) according to ASTM-G16. Loss in thickness over the exposure period or plotted as mass loss per unit area versus time will also be determined.

Subtask 7. Dry Oxidation Tests

Subtask 7A.   Atmospheric and Dry Oxidation Studies (G-54):* This practice will help us in determination of preliminary information on the growth, scaling, micro structural characteristics of an oxide on the surface and corrosion under isothermal conditions in still air. The ASTM procedure involves exposure of steel samples to temperatures at about 50-150°C for a certain time. Direct comparison of different materials at a constant temperature or the effect of temperature on a given material may be investigated, as oxidation is a dynamic time and temperature dependent process. The relative resistances of materials to oxidation at constant temperature should, therefore, be determined over at least three time periods. For these dry oxidation studies ASTM standard G-54 (dry oxidation) is applied. References will be made related to ASTM standards G-50, G-101, and G-31.

Subtask 7B. Thermogravimetric analyses (TGA): In the case of alloys such as SS 316L, and related Commercial SS Rock Bolts, and other alloys including C-22 are proposed for thermogravimetric analyses (TGA).
Experimental Method (G54):
The oxidation characteristics of some alloy types may be affected by humidity variations. It is thus important to include reference standards in each test series or to expose all materials to be compared at the same time. The specimens and their support are charged into the furnace operating at the test temperature and allowed to heat. After

this, samples will be taken out of the furnace and cleaned to remove the oxide formed on the surface in accordance with ASTM G-1 for the corrosion studies. The amount oxides formed on the surface can be determined by the "weight loss" measurement. Metallographic examination of the sample can also be done to determine the oxide penetration.

Following parameters will be determined from these studies:

Mass change per unit area:

The mass change per unit area (C) is given by the following Eq.20:

$$C = [(W_F - W_O)/A] \tag{20}$$

C = Mass change per unit area, g/m^2, W_F = Final Mass g, W_O = Original Mass, g and

A   = Original surface area, m^2.

The Depth of Attack (D) is given by the following Eq. 21:

$$D = [(T_O - T_m)/2] \tag{21}$$

D = Total depth of attack, T_O = Original thickness mm,

T_m = Remaining good metal, mm.

Thermogravimetric analysis (TGA) will be performed for accurate oxidation tests, and possibly to obtain rates of oxidation, using TA instruments TGA already available at UNR in Prof. Chandra's laboratory to compare the weight loss measurements in G-54 study.

Subtask 8: Characterization

There are 5 types of characterization tests that will be used on as-needed basis in this project. These are listed below.

Task 8A Metallography

Metallographic specimens were machined out of LCS and MCS into disk shapes and were mounted in epoxy resin using metallographic rubber molds. The molds were removed after the mix became firm around specimens and they were grinded until the steel surfaces were exposed. Then they manually ground with successive finer SiC emery papers to the 600-grit finish. Then they were polished manually using metallographic polishing cloth (Buehler microcloth) with successive finer alumina powders to the 0.05-micron finish. Then the polished specimen surfaces were degreased with acetone, and washed with tap water, and rinsed with distilled de-ionized water. The specimens were etched by dipping them into a 5% Nital enchant. LCS and MCS were required 20 and 10 seconds respectively, in order to achieve properly etched surfaces for metallographic evaluation.

Subtask 8B Scanning Electron Microscopy and Energy Dispersive X-Ray Study

The change in the corrosion morphology of rock bolts as a result of incorporation of the above ions in the electrolyte, and also the change in electrode potential, were observed by SEM in the secondary-electron imaging mode. The concentration of ions at the localized pit was analyzed by Energy Dispersive X-Ray Analysis (EDX) operated at a voltage of 20keV.

Subtask 8C Transmission Electron Microscopy

Transmission Electron Microscopy (TEM) will also be used to determine changes in the morphology of the steels as a result of the corrosion process at either Lawrence Berkeley Laboratory or Argonne National Laboratory/University of Illinois facilities, depending on the availability. We have just received a grant form NSF in our department for TEM but this going to take 6 to 8 months before it is functional.

Subtask 8D X-Ray Diffraction Analysis

Bulk phase changes as well as thick corrosion products (micrometer range films) may be characterized with x-ray diffraction analyses. The methods are commonly known, and we have several years of experience in this field to conduct this research.   We are proposing some equipment upgrade for the second year of the research.

Subtask 8E X-Ray Photoelectron Spectroscopy

 The XPS or ESCA analysis will be performed on rock bolt surfaces by additions of different ions using a Physical Electronics Quantum 2000 scanning ESCA microprobe available at the PNNL (Richland WA) user facility. The

samples are polished using SiC paper and passivated at pre-passive and passive potentials for 30 minutes. The change in chemical composition is analyzed using XPS, giving the surface depth profiles of the corrosion products.

Electrochemical studies will be carried out in our laboratory using simulated YM water demonstrated that the carbon steel underwent active, prepassive, passive and pitting behavior and also the passivation tendency was much pronounced when the ionic concentration was increased. Since the YM water contains HCO_3^- and SiO_2^- anions as an important passivating species and aggressive ions such as Cl^-, we also conducted some preliminary studies showing the synergistic effect of these anions on the passivation behavior of carbon steel and the oxide film changes that caused the increase in passivation tendency was analyzed using XPS and the results are interesting. In the similar way, we would like to carry out XPS analyses to analyze the structure and composition of the corrosion product film for the samples used for EIS and shear tested samples. The nature of various species with respect to thickness will be analyzed by depth profile analysis by sputtering the oxide film. The structure and composition of the passive film using X-ray Photoelectron Spectroscopy (XPS) will be studied. Following information will be obtained from the XPS spectra. We have made initial contact at Pacific Northwest National Laboratory (PNNL) to carry XPS experiments. Test sample from potentiodynamic scans under corroded conditions using different YM water concentration, and temperature will be examined at PNNL. Procedures are already outlined.

III.2: Quality Control and Data Recording, Reduction and Reporting Methods

The primary sources of error relating to this SIP will come from inaccuracies in recording equipment. All equipment used will therefore be calibrated and checked regularly to insure the validity of all data collected. Technical precision and representativeness of the tests to be performed is discussed in the descriptions of each subtask, in Section III.1.

All numeric raw data for this project will be controlled electronically. All electronic data will be protected in accordance with QAP-3.1, Control of Electronic Data.

All raw data for this project will be collected through laboratory acquisition. The data will be maintained electronically and its acquisition recorded in the scientific notebook. All data will be inspected visually for errors upon collection. Electronic transfer of data will be done in ".zip" format, which contains checksum algorithms to determine if any errors were introduced during the file transfer process.

Data reduction will primarily be done to reduce electrochemical data to corrosion rates. All data reduction will be recorded in the Scientific Notebook along with reference to the electronic data generated. All reduced data generated will be submitted to the TDA in accordance with QAP-3.6.

Subcontractors will be used for the collection of corrosion rates through salt spray tests and cyclic humidity tests. Outside calibration services will be performed with Bechtel Nevada. No models are currently planned for this task. These procurements will be obtained in accordance with QAP-3.0 and QAP-7.0.

IV Control of Samples

The sample used for this task will be metal alloys that are the subject of the corrosion studies.

Samples will be obtained by qualified vendors whenever possible and tracked in accordance with QAP 3.0 and QAP. 8.2.

Samples will be stored in a locked cabinet with limited access. A fire rated cabinet will be obtained for this purpose to better protect the samples. Samples will be registered with the Sample Management Facility if tracking becomes necessary. All samples will also be uniquely marked with a tag or engraving and a current inventory will be maintained with status of all samples.

When it is not possible to obtain samples from a qualified vendor, chemical and metallurgical analyses will be performed to verify the state of the sample in accordance with QAP 3.0.

V. Implementing Procedures and Standards

V.1 Standards

The following ASTM standards will be adapted for the development of IPs:

- A36 Standard specification for carbon structural steels
- A540 Standard Specifications for alloy- steel Bolting Material for special applications
- A194 Standard Specification for alloy steel Nuts for Bolts for High Pressure or High Temperature Service
- A490 Standard Specification for Heat Treated steel structural Bolts, 150 ksi Minimum tensile strength
- A307 Standard Specifications for Carbon steel Bolts and studs, 60000-psi tensile Strength
- A167-99 Standard Specification for Stainless and Heat-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip
- A176-99 Standard Specification for Stainless and Heat-Resisting Chromium Steel Plate, Sheet, and Strip
- A262-98 Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels
- A276-00 Standard Specification for Stainless Steel Bars and Shapes
- A370-97a Standard Test Methods and Definitions for Mechanical Testing of Steel Products
- A572 Specification for High-Strength Low-alloy Columbium-Vanadium Structural Steel
- B117-97 Standard Practice for Operating Salt Spray (Fog) Apparatus
- F-432-95 Standard Specifications for the Roof and Rock Bolts and Accessories
- G1-90 (1999) Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
- G3-89 (1999) Standard Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
- G5-94 Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements
- G15-99b Standard Terminology Relating to Corrosion and Corrosion Testing
- G16-95 (1999) Standard Guide for Applying Statistics to Analysis of Corrosion Data
- G28-97 Standard Test Methods of Detecting Susceptibility to Intergranular Corrosion in Wrought, Nickel-Rich, Chromium-Bearing Alloys
- G30-97 Standard Practice for Making and Using U-Bend Stress-Corrosion Test Specimens
- G31-72 (1999) Standard Practice for Laboratory Immersion Corrosion Testing of Metals
- G36-94 Standard Practice for Evaluating Stress-Corrosion-Cracking Resistance of Metals and Alloys in a Boiling Magnesium Chloride Solution
- G38-73 (1995) Standard Practice for Making and Using C-Ring Stress-Corrosion Test Specimens
- G39-99 Standard Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens
- G48-99a Standard Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution
- G49-85 (1995) Standard Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens
- G50-76 Standard Practice for Conducting Atmospheric Corrosion Tests on Metals
- G54-84 Standard Practice for Oxidation Testing
- G58-85 (1999) Standard Practice for Preparation of Stress-Corrosion Test Specimens for Weldments
- G59-97 Standard Practice for Conducting Potentiodynamic Polarization Resistance Measurements
- G60-95 Standard Test Method for Conducting Cyclic Humidity Tests
- G61-86 (1998) Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys
- G71-81 (1998) e1 Standard Guide for Conducting and Evaluating Galvanic Corrosion Tests in Electrolytes
- G78-95 Standard Guide for Crevice Corrosion Testing of Iron-Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments
- G85-98 Standard Practice for Modified Salt Spray (Fog) Testing
- G100-89 (1999) Standard Test Method for Conducting Cyclic Galvanostatic Polarization
- G101-97 Standard Guide for Atmospheric Corrosion Resistance of Low-Alloy Steels
- G102-89 (1999) Standard Practice for Calculation of Corrosion Rates and Related Information from Electrochemical Measurements
- G106-89 (1999) Standard Practice for Verification of Algorithm and Equipment for Electrochemical Impedance Measurements
- G107-95 Standard Guide for Formats for Collection and Compilation of Corrosion Data for Metals for Computerized Database Input

- G108-94 (1999) Standard Test Method for Electrochemical Reactivation (EPR) for Detecting Sensitization of AISI Type 304 and 304L Stainless Steels
- G123-96 Standard Test Method for Evaluating Stress-Corrosion Cracking of Stainless Alloys with Different Nickel Content in Boiling Acidified Sodium Chloride Solution
- G129-95 Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking
- G142 Standard Practice for Hydrogen Embrittlement Cracking
- G148-97 Standard Practice for Evaluation of Hydrogen Uptake, Permeation, and Transport in Metals by an Electrochemical Technique

V.2 Implementing Procedures

The following current Implementing Procedures (IP) will be followed as appropriate. Other IP shall be developed as deemed necessary.

- IPLV-003 "Analytical and Top Loading Balance Use"

VI Equipment

The following equipment used will be calibrated by Bechtel Nevada.

EQUIPMENT	MODEL	S/N
Omega Type J Thermocouple	5TC-TT-J-30-36	TH-1
Omega Type J Thermocouple	5TC-TT-J-30-36	TH-2
Omega Type J Thermocouple	5TC-TT-J-30-36	TH-3
Kessler Glass Thermometer	TTL1MM	418308
Kessler Glass Thermometer	TTL1MM	259084
Kessler Glass Thermometer	TTL1MM	259134
Fluke Digital Multimeter	87 III	74370382
Omega Digital Thermometer	HH23	T-106739
Fluke Digital Thermometer	54 II	79440064
Mitutoyo Digital Caliper	CD-S8"C	1574
Econo Temperature Controller	32A	12125-14
Cole-Parmer Temperature Controller	89810-00	J1527

The following equipment to be used will be calibrated on site by Bechtel Nevada.

EQUIPMENT	MODEL	S/N
Epsilon Extensometer	3542-0064	025ST
United Calibrations Force Test Machine	STM-10-ES	0302515
TA Instruments Thermo Gravimetric Analyzer	TGAQ500	1.143265
Interface Load Cell	2440BXM-10K	101560A
Mettler Balance	AE 20	N28015

The following equipment will be checked using in house calibration procedures.

MFR/EQUIPMENT	MODEL	S/N	UNR ID
Gamry/ Potentiostat	PC4/750	1230127	Pstat 1
Gamry/ Potentiostat	PC4/750	1230126	Pstat 2

Any equipment acquired under this task for use with quality affecting work will also require calibration including the MTS nano indenter mentioned above.

No equipment for this task is in a public location.

VII Hold Points and Decision Points

There are no hold points or decision points for this task.

VIII Records, Reports and Submittals

The following submittals are planned for this task noted with the time and frequency necessary.

Project level:
    Scientific Investigation Plan   One time, revised as needed
    Implementing Procedures   As needed
    Personnel Training Documentation   As needed
Operationally
    Scientific Notebook entries   At time of data collection
    Calibrations   As needed, usually annually
Reporting
    Quarterly reports to DOE, Non QA   Quarterly
    Scientific Notebook Reviews   As needed, and at project close out
    Final technical report to DOE   At project close out

The following QA records will be generated for this task.
    Scientific Investigation Plan
    Implementing Procedures developed as needed
    Scientific Notebooks recording work as it is completed
    Calibration reports including calibration checks and end-point calibration reports
    Final Technical Report to be submitted to DOE.

These records will be protected and transmitted in accordance with QAP 17.0 "Quality Assurance Records."

IX Verification and Reviews

Scientific Notebooks generated by this task will receive technical and QA reviews as needed and at the close of the task. Any technical reports generated by this task will be reviewed in accordance with QAP 3.4

X Software

We do not at present intend to use any software packages or models requiring qualification.

XI Interface Controls

The Principal Investigator for this project is Dr. Dhanesh Chandra. He will be responsible for overseeing the project and assuring adequate work progress as well as maintaining the scientific validity of the project. Drs. Jaak Daemen and Raul Rebak will provide technical assistance and assist in the performance of technical reviews. The DOE Technical Task Representative for this project is Jaime Gonzalez.

The majority of laboratory work will be performed at the University of Nevada, Reno. Any work performed outside of the facilities will be done either through a qualified supplier or under the supervision of an investigator for this task. The UCCSN QA program will provide training as required. All work planned and all qualified procurements are subject to review and/or verification by the UCCSN QA manager.

Technical data from the previous task may be used, detailed in UCCSN Technical Report: TR-03-017. Also, the water composition used for electrochemical tests will be based on compositions supplied by the DOE and be accompanied by an associated Data Tracking Number (DTN).

XII Schedule

We will produce data for corrosion rates, in the 4 years and 3 months, based on electrochemistry as well conventional ASTM "immersion type tests," SCC/HE, hydrogen permeation in form of reports, following QA procedures, on the following:

1. Corrosion test results (corrosion rates) by Potentiodynamic (Short Term) Tests using YM waters on 5 alloys that will be selected from the screening tests; tests include effect of temperature and ionic concentration.
2. Immersion (Long-Term) Corrosion test results by Potentiodynamic Tests on the Alloys using YM waters on the 5 alloys.
   These will be cyclic humidity tests, fog tests, and immersion tests.
3. Stress Corrosion Cracking and Hydrogen Embrittlement tests, with different strain rates, on the 5 alloys at two different temperatures.
4. Hydrogen Permeation tests on the 5 alloys at two different temperatures.
5. Dry Oxidation tests (ASTM and TGA tests) on all the alloys mentioned above.
6. Characterization of corrosion products microstructure and crystal, where necessary, by SEM,TEM, XPS, Metallography.
7. Deliver Quarterly reports and a Final report at the end of 4 years 3 months.

Subtask				FY04	Q_1	Q_2	Q_3	FY05	Q_1	Q_2	Q_3	FY06	Q_1	Q_2	Q_3	FY07	Q_1	Q_2	Q_3	FY08
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Reports				x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x	Final

XIII References

[1] J. Daeman, D.Chandra and D.A. Jones "Long term Drift stability of rock bolts and steel sets in YM repository environment" SIP-UNR-018 (1991).

[2] D. Chandra, J. Daemen, A. Venugopal, A. Yilmaz, V. Deodeshmukh, Joshua Lamb, "Final Report to YM project, " Corrosion Behavior of Rock bolts and Steel Seats, (Nov 2003).

[3] V. Deodeshmukh, "Electrochemical Polarization and Hydrogen Permeation behavior of carbon steels proposed for Yucca Mountain Repository, M.S. Thesis, University of Nevada, Reno (Advisor: D. Chandra, December 2002.

[4] A. Yilmaz, "Degradation and Failure Susceptibility of Carbon Steels in Simulated Yucca Mountain Nuclear Repository Environment," Ph.D. Dissertation, University of Nevada, Reno (Advisor: D. Chandra), August 2003.

[5] McNabb, A and Foster, P.K., Metallurgical Society of the American Institute for Mining, Metallurgical and Petroleum Engineers, New York, 227 (1963), p618.

[6] G.M. Pressouyre, R. Blondeau, G. Primon and L. Cadiou, (eds C.G. Interrante and G.M. Pressouyre)- Proceedings of the First International Conference on Current Solutions to Hydrogen Problems in Steels, ASM, Washington, D.C., p.18 (1982).