UCRL-ID-131585

Stress Corrosion Cracking Tests Using Double-Cantilever-Beam Specimens

Ajit Roy

October 25, 1996

Lawrence Livermore National Laboratory

Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

Available to DOE and DOE contractors from the
Office of Scientific and Technical Information
P.O. Box 62, Oak Ridge, TN 37831
Prices available from (423) 576-8401

Available to the public from the
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Rd.,
Springfield, VA 22161

UCRL-ID 131585

University of California Lawrence Livermore National Laboratory **YUCCA MOUNTAIN PROJECT** **Activity Plan**	No.: E-20-56 Revision: 0 Change Notice: AP E-20-56-0-1 Page: 1 of 13
Subject: Stress Corrosion Cracking Tests Using Double-Cantilever-Beam Specimens	AUTHOR: Ajit Roy

Training Required: Yes ☐ No ☐

Comments: N/A

REVISION HISTORY

Rev. No.	CN No.	Effective Date	Description of Revision/CN
0		09/30/96	Original issue.
0	AP E-20-56-0-1	10/25/96	Administrative changes to correct typographical errors. Affects Title Page and pages 2 and 3.

APPROVALS:

W. L. Clarke — 10/22/96

W.L. Clarke, CRWMS LLNL Manager — Date

R. E. Monks — 21 Oct '96

R.E. Monks, M&O LLNL Quality Assurance Manager — Date

R. Daniel McCright — 18 Oct 1996

R.D. McCright, Technical Area Leader — Date

Stress Corrosion Cracking Tests Using Double-Cantilever-Beam Specimens

Activity Plan E-20-56

YMP WBS Element 1.2.2.5.1

LLNL-YMP Metal Barrier Selection and Testing

Lawrence Livermore National Laboratory

Revision 0

Effective Date: 09/30/96

TABLE OF CONTENTS

Page

LIST OF TABLES

LIST OF FIGURES

Page

1.0 SCIENTIFIC INVESTIGATION PLAN

This activity plan is prepared in accordance with Lawrence Livermore National Laboratory (LLNL) Yucca Mountain Project procedure 033-YMP-QP 3.0, "Scientific Investigation Control." This plan is written for activity E-20-56, entitled "SCC Investigations using Self-Loaded Specimens," which is a part of the Scientific Investigation Plan (SIP) "Metal Barrier Selection and Testing" (CN SIP-CM-01, Rev. 3), WBS #1.2.2.5.1.

1.1 Activity Identity

The activity E-20-56 entitled "Stress Corrosion Cracking Tests Using Double-Cantilever-Beam Specimens," is a newly-developed activity, and will involve short-term Abiotic Laboratory Corrosion Testing described in the SIP "Metal Barrier Selection and Testing." As described in CN SIP-CM-01, Rev. 3, this activity will evaluate the stress corrosion cracking (SCC) susceptibility of candidate corrosion-resistant waste package container materials using fracture-mechanics-based double-cantilever-beam (DCB) specimens.

1.2 Responsibilities

Key personnel responsible for performing the work in this activity are:

Technical Area Leader: Engineered Barrier Materials	Dr. R.D. McCright
Lead Principal Investigator: Electrochemical and Fracture Mechanics Testing	Dr. A.K. Roy
Scientific Associate	Mr. J. C. Estill

2.0 SCOPE, PURPOSE, AND OBJECTIVES

Although a wide variety of degradation modes can occur in aqueous environments for corrosion-resistant metallic materials, localized corrosion such as pitting corrosion, crevice corrosion, SCC, and hydrogen embrittlement (HE) is considered to be the primary mode. The evaluation of the susceptibility of candidate corrosion-resistant container materials to pitting and crevice corrosion is well underway using electrochemical polarization techniques described in the Activity Plan E-20-43/44. The proposed activity (E-20-56) is aimed at evaluating the SCC behavior of these materials in susceptible environments using the linear-elastic-fracture-mechanics (LEFM) concept. The mechanical driving force for crack growth, or the stress distribution at the crack tip is quantified by the stress intensity factor, K, for the specific crack and loading geometry. The critical stress intensity factor for SCC, K_{ISCC}, for candidate materials will be evaluated in environments of interest, and their comparisons will be made to select the waste package inner container material having an optimum SCC resistance.

3.0 ACTIVITY DESCRIPTION

3.1 Fundamentals of SCC and LEFM

SCC is an environment-assisted cracking phenomenon resulting from the combined and synergistic interactions of tensile stress and a specific corrosive environment.

Environments causing SCC are usually aqueous, and can be either condensed layers of moisture or bulk solutions. HE is also a form of environment-induced failure that results most often from the combined action of hydrogen , and residual or applied tensile stress. While several mechanisms of SCC and HE have been proposed based on numerous parametric studies, not a single unique mechanism has been widely accepted, since they all contain elements of speculation and none has been demonstrated beyond a doubt.

The LEFM approach assumes that cracks are either initially present or are initiated early in a structural component, and that the structural failure results from the growth of these cracks by SCC. With the advent of LEFM, there has been a trend towards quantitatively relating the crack growth rates to the mechanical driving force under various environmental conditions. The mechanical driving force for crack growth is considered to be given by the crack-tip stress intensity factor (K_I) defined by linear elasticity.

Generally, a notched specimen[1] is used to evaluate the crack growth rate of engineering materials resulting from SCC. In terms of specimen size, there are two distinctly separate requirements: one pertaining to the applicability of LEFM, and the other related to the condition of constraint at the crack-tip (i.e. plane strain versus plane stress). The first one involves the minimum size of the crack and other planar dimensions of the specimens that are needed to satisfy the assumptions of limited plasticity. The second one is concerned with the degree of relief of constraint in the thickness direction by localized plastic deformation (yielding) at the crack-tip. Both these requirements relate to the size of the crack-tip plastic zone. It has become convenient[1] to use the parameter $(K_I / \sigma_{YS})^2$ as a measure of the size of the plastic zone at the crack-tip, where σ_{YS} is the uniaxial tensile yield strength. Fracture toughness data suggest that in order to satisfy the assumption of limited plasticity, the minimum crack length (a) and the specimen thickness (B) should be equal to or greater than $2.5\ (K_I / \sigma_{YS})^2$.

3.2 DCB Test Technique

The double-cantilever-beam (DCB) test is a crack-arrest type of fracture mechanics test for measuring the resistance of metallic materials to propagation of SCC, expressed in terms of critical stress intensity for SCC, K_{ISCC}. The susceptibility to SCC will be evaluated by stressing a DCB specimen by inserting a double taper wedge, as shown in Figure 1. The wedge shall be made of the same material as the test material. DCB specimens will be fabricated from heat-treated bar materials of interest by an outside vendor. Side grooves having a semicircular cross section (rather than the triangular cross section shown in Figure 1) will be cut into both sides of the specimen to a specified depth to restrict crack growth to a single plane. Machining of the side grooves must be done carefully to avoid overheating and cold working. Specimens will be tested in the as-received condition without any additional thermal treatment.

Prior to insertion of the wedge, the DCB specimen will be fatigue-precracked using an Instron Servohydraulic testing machine having a 55 kip load-cell. To avoid residual compressive stress, the peak K_I during precracking should not exceed 70% of the expected initial K_I imparted by the wedge. The thickness of the wedge will be determined by the desired amount of arm-displacement of the DCB specimen.

A total of eight specimens per candidate alloy will be tested. Duplicate specimens of each alloy will be vertically placed in a rack made of an insulating material, which will then be inserted into a glass chamber containing the test solution. The test specimens will be totally immersed in the deaerated solution. Deaeration will be done by bubbling the test solution with nitrogen for one hour. The desired solution temperature will be attained by inserting the glass chamber inside a heating mantle. A thermocouple will be used in each test chamber to measure temperature at the desired value within ± 5°C. A pyrex condenser will be fitted to one port of the gas-tight glass lid to capture evaporated water and return it to the test chamber. A metallic clamp will be used to hold the lid on top of the glass chamber. Solution pH will be measured at room temperature both before and after each test.

Testing will be conducted for periods of one, two, four, and six months. At the end of each test, all specimens will be visually examined, followed by an optical microscopic examination. Upon completion of each test, the equilibrium wedge load will be measured by applying a separating force to the specimen arms in an Instron Servohydraulic testing machine. The specimens will then be split open, and the crack length will be measured on both faces of the specimen. Knowing the specimen dimensions, the equilibrium wedge load, and the crack length, the stress intensity for SCC can be computed by using the following equation[2] :

$$K_{ISCC} = \frac{Pa\,(2\sqrt{3} + 2.38\ h/a)\,(B/B_n)^{1/\sqrt{3}}}{Bh^{3/2}}$$

where:

K_{ISCC}	= Threshold stress intensity for SCC
P	= Equilibrium wedge load, measured in the loading plane
a	= Crack length
h	= Height of each arm
B	= Specimen thickness
B_n	= Web thickness

Measuring crack length at the conclusion of each test, crack growth rate (da/dt) can be computed and can be plotted as a function of K_{ISCC}. The morphology of cracking in each specimen will be studied using the scanning electron microscope.

3.3 Test Materials

A list of corrosion-resistant alloys recommended for testing is shown in Table 1. This includes iron-nickel-chromium-molybdenum Alloys 825, G-3 and G-30; nickel-chromium-molybdenum (Ni-Cr-Mo) Alloys 625, C-4 and C-22; and titanium-base alloy Ti Gr-12. Alloys G-3 and G-30 are comparable in composition to Alloy 825, but have somewhat higher alloy content, for enhanced resistance to localized corrosion. Alloys 625, C-4 and C-22 have been identified for testing because of their high-temperature stability and superior overall corrosion resistance compared to other Ni-Cr-Mo alloys available today. Ti Gr-12 has been selected in view of its outstanding corrosion resistance, and its useful combination of low density and high strength. The list of materials identified in Table 1 may be modified later as work progresses in the Waste Package Design and Waste Package Materials areas.

3.4 Test Environments

Although the groundwater in the vicinity of the proposed repository (Well J-13) is known to have a near-neutral to slightly alkaline pH, and to be benign to corrosion-resistant materials, the precise environment surrounding the waste packages inside the repository is yet to be determined. The results of recent electrochemical study performed at LLNL showed that some of the candidate corrosion-resistant alloys may become susceptible to pitting corrosion in acidified (pH~2-3), concentrated salt solution at 90°C. It is conceivable that such conditions could be present in the proposed repository under some scenarios. Therefore, initial SCC testing will be performed in a similar environment. Test solutions will be made from distilled water and reagent grade chemicals, following standard laboratory practices. The test environment may be modified later to study the effects of variables such as pH, chloride ion concentration, other ionic species, and temperature on cracking tendency of materials of interest.

3.5 Technical and Readiness Reviews

No additional formal Readiness Review (QP 2.6) is planned for this activity. No formal technical review (QP 2.4) is planned at the completion of the present activity. However, depending on the progress of technical work in this activity and related ones, a technical review may be held to review the adequacy of the SCC testing for making long-term performance predictions.

3.6 Hold Points

The operation of the testing facility will be monitored on a continuous basis by the Lead Principal Investigator to ensure that the work is proceeding according to plan. If significant unanticipated problems arise, the Lead Principal Investigator will inform the Technical Area Leader. A joint decision will be made about the future course of action.

The progress of testing will be reported to the Technical Area Leader in periodic reports. If substantial changes in project scope require that experimental work change significantly in direction, the Technical Area Leader will communicate this to the Lead Principal Investigator in writing. No formal hold points or decision points will be designated.

3.7 Special Training/Qualification Requirements

Qualifications of the Principal Investigator(s) and technicians are specified by the Technical Area Leader in accordance with 033-YMP-QP-2.10, "Qualification of Personnel." A Principal Investigator (PI) shall have a Ph.D. or equivalent in materials science, metallurgy, metallurgical engineering, corrosion engineering, or related field. Technical support staff shall have experience in electrochemical, corrosion, mechanical, and electrical instrumentation and techniques. Only personnel trained to appropriate quality procedures and any other procedures of the Yucca Mountain Site Characterization Project will be allowed to participate in these activities. Assignment of personnel may change with time. Names of personnel authorized to perform the experimental work in this activity will be given in the appropriate scientific notebook. The current position descriptions, management certifications, and QA training records should be consulted for more details.

3.8 Quality Assurance Program

This activity to be conducted in support of "Metal Barrier Selection and Testing" as described in SIP-CM-01, Rev. 3. This study has been determined to be a quality affecting activity, subject to the provisions of all applicable quality assurance procedures.

In particular, certain parts of the QP manual that will be followed are:

1. Measurement will be performed, and test equipment (M&TE) will be calibrated as specified in Procedure 033-YMP-QP 12.0, "Control of Measuring & Test Equipment." See also Section 4.0 in this activity plan.

2. Test specimens will be procured as specified in Procedure 033-YMP-QP 4.0, "Procurement Document Control," and controlled as specified in Procedure 033-YMP-QP 8.0, "Identification and Control of Items, Samples, and Data."

3. Collected data will be controlled as specified in Procedure 033-YMP-QP 8.0, "Identification and Control of Items, Samples, and Data."

4. Scientific notebooks will be maintained as specified in Procedure 033-YMP-QP 3.4, "Scientific Notebooks."

5. Technical reports will be prepared, reviewed, and approved as specified in Procedure 033-YMP-QP 3.3, "Review of Technical Publications." Technical data generated in this activity will be processed as specified in Procedure 033-YMP-QP 3.6, "Collection, Review and Submittal of Technical Data," according to the kind of data and the desired disposition of the information.

3.9 Activity Close-out

As with all other activities in the Metal Barriers task, the major reporting channel is through periodic revision of the Engineered Materials Characterization Report or EMCR, which is Activity E-20-39, in SIP-CM-01 Rev. 3. Supporting documentation such as scientific notebooks and technical report review comments will be retained by the appropriate individual (PI or technical support personnel) until the document package is transferred to the LLNL/YMP Local Records Center at the conclusion of these activities. Many of these records are transferred periodically as record segments so that the final records package of this activity is compiled over a period of time. QA records will be transmitted as described in Procedure 033-YMP-QP-17.0, "Quality Assurance Records."

No additional or special activity close-outs are planned.

4.0 PRECISION AND ACCURACY

4.1 Calibration Requirements

4.1.1 Test Solutions

Test solutions are made by weighing out a quantity of reagent and dissolving this quantity in the appropriate volume of water. The precision of the laboratory balance or other weighing device is not a critical issue, since the solution composition is a target, not a control. A commercial grade electronic balance, capable of weighing up to two decimal places (a hundredth of a gram) will provide more than adequate precision for this activity when operated in conformance with the manufacturer's instructions. Should an anomaly be suspected in the results, then the operation of the balance can be confirmed by tarring the balance to zero and confirming the weight of a known volume of water. An accuracy within 0.1% will provide adequate performance.

Solution pH is regularly measured in characterizing the test environment. Although a target pH is usually sought, the purpose of the measurement is not so much for control of the pH as it is to describe the environment. Standard laboratory pH meters or even indicator papers have sufficient accuracy for this purpose, since only accuracy to the integer value is needed. If a pH meter is used, it will be user-calibrated with use of known buffer solutions just prior to use and following the manufacturer's recommended procedure given in the operating manual. Any doubt about the pH meter or indicator paper is readily resolved by measuring standard pH buffer solutions.

4.1.2 <u>Other</u>

Load-cells used in fatigue precracking of the DCB specimens will be calibrated with dead weights at the beginning of each test series. Also, the thermocouples used in measuring the temperature of the test-cells will be calibrated at a qualified testing laboratory.

4.2 Sources of uncertainty and error to be controlled and measured

The extent of cracking in susceptible alloys may vary from specimen to specimen due to random variations in alloy composition, alloy microstructure, specimen surface micro-features, and other factors. Although their effects are usually small, duplicate specimens will be tested to compare their crack-growth. It is expected that the calibrations and replications planned will control the effects of any conditions that could adversely affect results.

5.0 IN-PROGRESS DOCUMENTATION

Documentation to be generated during the conduct of this activity will include scientific notebooks, and may also include data record sheets, raw data, progress reports, and the final report. Scientific notebooks are controlled and maintained according to procedure 033-YMP-QP 3.4, "Scientific Notebooks." Test specimens will be controlled and maintained according to procedure 033-YMP-QP 8.0, "Identification and Control of Items, Samples, and Data." No Technical Implementing Procedures will be prepared for this activity.

Along with other technical activities in the Metallic Barriers Task, reporting of the results of this activity will occur on a regular and periodic basis as determined by the schedule of project deliverables. Also, the results will be reported as revisions to the EMCR. As appropriate, topical LLNL reports (UCRL series) will be prepared on parts of this activity. Interim reports may also be written if deemed appropriate. The report(s) will undergo technical review as specified in procedure 033-YMP-QP 3.3, "Review of Technical Publications and Data."

6.0 INTERFACES

The information obtained from this experimental activity will assist activities in the following technical areas, and copies of the written reports from this activity will be distributed to the individuals designated:

(1) Metal Barrier Selection and Testing (SIP-CM-01)
R. D. McCright, TAL, Engineered Barrier Materials

(2) Waste Package Performance Assessment Activities (SIP-PA-2)
W. Halsey, TAL, Performance Assessment

7.0 SCHEDULE

The current PACS budget and schedule should be consulted.

8.0 SPECIAL CASES

No subcontractors are involved in these activities.

9.0 REFERENCES

1. ASTM Designation: E 399 - 90, "Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials," American Society for Testing and Materials 1996 Book of Standards, volume 03.01, pp. 407-437, ASTM, Philadelphia (1996).

2. NACE Standard TM0177-90, Standard Test Method - Method D (Double-Cantilever-Beam Test), National Association of Corrosion Engineers, Item No. 53040, Houston (1990).

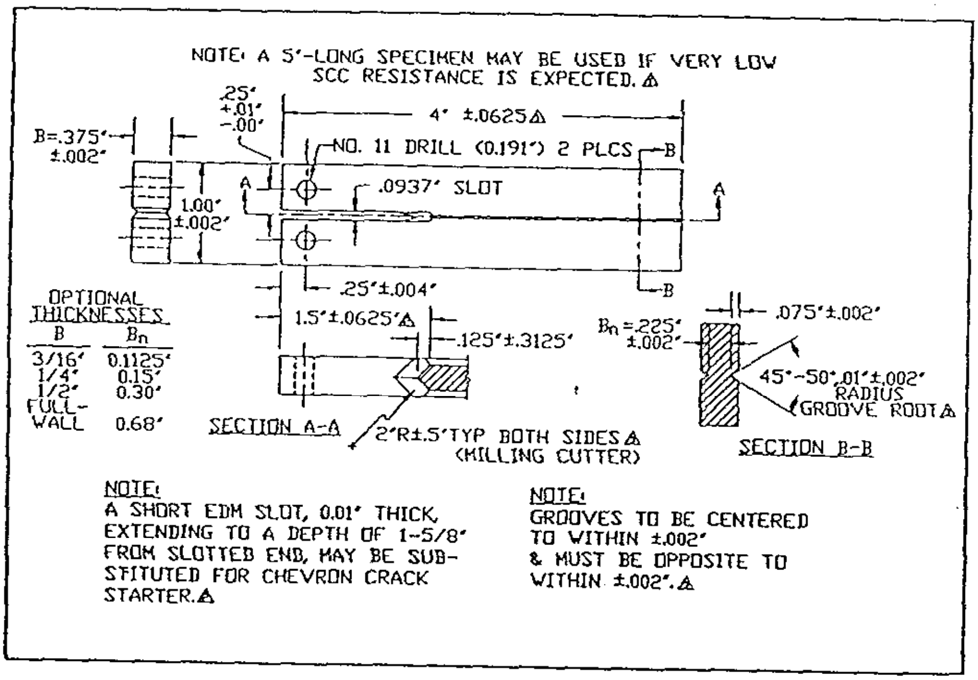
10.0 APPENDIX

There are no appendices.

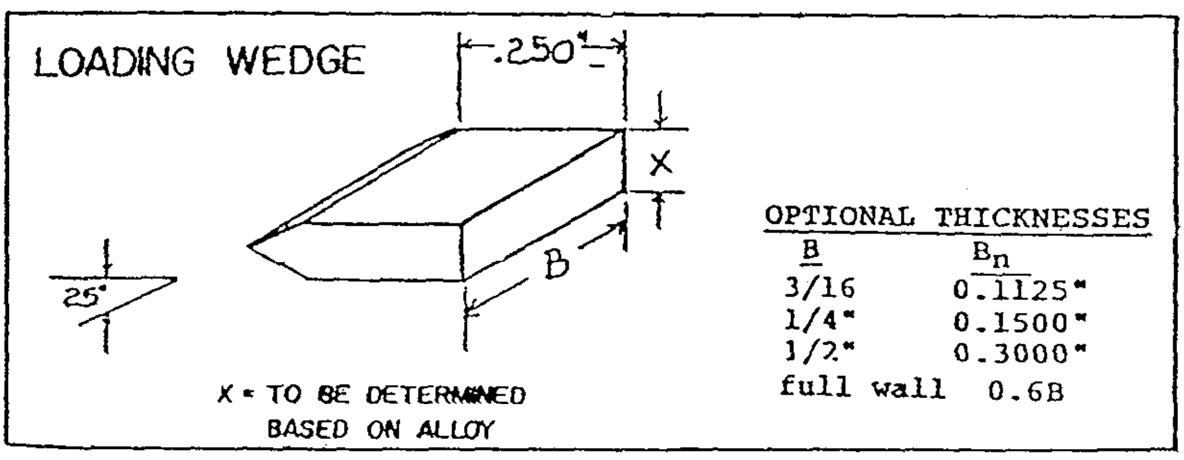
Table 1

List of Materials Recommended for Testing

Commercial Name	UNS Number	ASTM Number
Alloy 825, Incoloy 825	N08825	B 425
Hastelloy Alloy G-3	N06985	B 581
Hastelloy Alloy G-30	N06030	B 581
Alloy 625, Inconel 625	N06625	B 443
Hastelloy C-4, Alloy C-4	N06455	B 575
Hastelloy C-22, Alloy C-22	N06022	B 574
Titanium Grade-12	R53400	B 265 Grade 12



(a)



(b)

Figure 1. (a) DCB Specimen ; (b) Double Taper Wedge