PUL-5930

**Cover Sheet for a Hanford
Historical Document
Released for Public Availability**

Released 1994

**Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute**

DISCLAIMER

This is a historical document that is being released for public availability. This was made from the best available copy. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

UNCLASSIFIED

UNC NUCLEAR INDUSTRIES

A UNC RESOURCES Company

P.O. Box 490
Richland, Washington 99352PREPARED FOR THE
UNITED STATES DEPARTMENT OF ENERGY
UNDER CONTRACT NUMBER DE-AC06-76RL01857

Document No.

UNI-3994

Date

June 1986

Copy No.

(Reserved For Document Control Center Use Only)

Title

ZIRCONIUM PRESSURE TUBE TESTING:
TEST PROCEDURES
PRODUCTION ASSURANCE PROGRAM (PROJECT H-700)

Author

F. R. Zaloudek, PNL, for E. S. Ruff, UNC

DISTRIBUTION

NAME		BLDG/RM/MS	NAME		BLDG/RM/MS
1.	ER Gilbert	PSL/1410/3000	23.	EV Padgett	313/60B/300
2.	M Lewis	377/T2-1/300	24.	AK Park	3703/25/300
3.	EB Schwenk	314/102/300	25.	PC Payne	1101N/119/N2
4.	FR Zaloudek	PSL/1400/3000	26.	DL Renberger	1117N/13/100N
5.	JC Tobin	337/2S27/300	27.	ES Ruff (5 copies)	1155 Jadwin
6.	JD Bateman	313/6A/300	28.	WG Ruff	1117N/16/100N
7.	DC Boardman	3703/25/P1	29.	KV Scott	325/933/W-C38
8.	GM Burnett	1120 N/Bay/100N	30.	EL Shaber	3703//1300
9.	RA Cox	333/212B/300	31.	JW Sharp	333/216/300
10.	WM DeVelin	3703/20/300	32.	RH Snyder	1155 Jadwin
11.	LM Gonzalez	FED/423/700	33.	JW Stiffler	3703/11/300
12.	RE Gross	3703/23/P1	34.	DJ Trimble	325/938/300
13.	JA Hunter	FED/420B/700	35.	JE Werle	FED/420F/700
14.	JP Kester	FED/420A/700	36.	Project Files: H-700, H-703	1118N/39/B5
15.	LK Khosla	1145N/2/100N	37.	PQE File	325/12B/WC38
16.	EJ Krejci	1145N/3/100N	38.	UNC Files/UNC Records	FED/436/F11
17.	LP Leach	FED/418/700			
18.	HD Lenkersdorfer	1155 Jadwin			
19.	FS Metzger	1155 Jadwin			
20.	JJ McGuire	313/59/300			
21.	J Newell	1145N/6/100N			
22.	RJ Nicklas	FED/417/700			
ROUTE TO		PAYROLL NO.	LOCATION	FILES ROUTE DATE	SIGNATURE AND DATE

UNCLASSIFIED

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

REFERENCE

COPY

BM-5100-184 (4-85)

ONLY

UNC NUCLEAR INDUSTRIES, INC.

RICHLAND, WA. 99352

NOTICE

THIS REPORT WAS PREPARED AS AN ACCOUNT OF WORK SPONSORED BY THE UNITED STATES GOVERNMENT. NEITHER THE UNITED STATES NOR THE UNITED STATES DEPARTMENT OF ENERGY, NOR ANY OF THEIR EMPLOYEES, NOR ANY OF THEIR CONTRACTORS, SUBCONTRACTORS, OR THEIR EMPLOYEES, MAKES ANY WARRANTY, EXPRESS OR IMPLIED, OR ASSUMES ANY LEGAL LIABILITY OR RESPONSIBILITY FOR THE ACCURACY, COMPLETENESS OR USEFULNESS OF ANY INFORMATION, APPARATUS, PRODUCT OR PROCESS DISCLOSED, OR REPRESENTS THAT ITS USE WOULD NOT INFRINGE PRIVATELY OWNED RIGHTS.

DISCLAIMER

MASTER

THIS REPORT WAS PREPARED FOR USE WITHIN UNC NUCLEAR INDUSTRIES, INC., IN THE COURSE OF WORK UNDER SUPPLEMENTAL AGREEMENT TO THE UNITED STATES DEPARTMENT OF ENERGY CONTRACT DE-AC06-76RL01857. ANY VIEWS OR OPINIONS EXPRESSED IN THE REPORT ARE THOSE OF THE AUTHOR ONLY. THIS REPORT IS SUBJECT TO REVISION UPON COLLECTION OF ADDITIONAL DATA.

ZIRCONIUM PRESSURE TUBE TESTING: TEST PROCEDURES

PRODUCTION ASSURANCE PROGRAM

ISSUED BY

BATTELLE, PACIFIC NORTHWEST LABORATORY

APPROVALS

Authors
(PNL)

F. R. Zaloudek
F. R. Zaloudek

Date 6/17/86

M. Lewis
M. Lewis

Date 6/17/86

Independent
Review
(UNC)

E. S. Ruff
E. S. Ruff

Date _____

Issuing
Manager
(UNC)

R. H. Snyder
R. H. Snyder

Date _____

Quality
Assurance
(UNC)

D. C. Board
D. C. Board

Date _____

Construction
Program
Manager
(UNC)

H. D. Lenkersdorfer
H. D. Lenkersdorfer

Date _____

ZIRCONIUM PRESSURE TUBE TESTING: TEST PROCEDURES
PRODUCTION ASSURANCE PROGRAM

F. R. Zaloudek
M. Lewis

June 1986

Prepared for
The U. S. Department of Energy
Under Contract DE-AC06-76 RLO-1830

Pacific Northwest Laboratory
Richland, Washington 99352

ACKNOWLEDGEMENTS

The authors wish to acknowledge the technical comments given by Eric L. Shaber, Ray E. Gross, John E. Tobin, Dennis J. Trimble, and Don C. Board of UNC Nuclear Industries, and John C. Tobin of the Hanford Engineering Development Laboratory.

CONTENTS

1.0 Introduction	1
2.0 TENSILE TESTING PROCEDURES	2
2.1 Applicable Standards and Specifications	2
2.2 General Description of Method	3
2.3 Sample Description	4
2.3.1 Sample Dimensions.....	4
2.3.2 Sampling Method	4
2.3.3 Sample Preparation	4
2.3.4 Sample Disposition	5
2.4 Test Equipment	5
2.4.1 Equipment Description	5
2.5 Procedure	6
2.5.1 Pre-Test Procedure	6
2.5.2 Test Procedures	7
2.5.3 Post-Test Procedures	8
2.5.4 Data Records	9
2.5.5 Witnesses	9
2.6 Data Analysis	10
2.6.1 Data Reduction Methodology	10
2.6.2 Data Reported	10
3.0 BURST TEST PROCEDURES	14
3.1 Applicable Standards and Specifications	14
3.2 General Description of Method	14
3.3 Sample Description	15
3.3.1 Sample Dimensions	15
3.3.2 Sampling Method	16
3.3.3 Sample Preparation	16
3.3.4 Sample Disposition	17
3.4 Test Equipment	17
3.4.1 Equipment Description	17
3.5 Procedures.....	20
3.5.1 Pre-Test Procedures	20
3.5.2 Test Procedures	21

3.5.3 Post-Test Procedures	23
3.5.4 Data Records	24
3.5.5 Witnesses	24
3.6 Data Analysis	24
3.6.1 Data Reduction Methodology	24
3.6.2 Data Reported	25
4.0 FRACTURE TOUGHNESS TESTING	27
4.1 Applicable Standards and Specifications	27
4.2 General Description of Method	27
4.3 Sample Description	29
4.3.1 Sample Dimensions	29
4.3.2 Sampling Method	29
4.3.3 Sample Preparation	29
4.3.4 Sample Disposition	30
4.4 Test Equipment	30
4.4.1 Equipment Description	30
4.5 Procedures.....	31
4.5.1 Pre-Test Procedures	31
4.5.2 Test Procedures	32
4.5.3 Post-Test Procedures	33
4.5.4 Data Records	33
4.5.5 Witnesses	33
4.6 Data Analysis	34
4.6.1 Data Reduction Methodology	34
4.6.2 Data Reported	36
5.0 CORROSION RATE TESTING	41
5.1 Applicable Standards and Specifications	41
5.2 General Description of Method	42
5.3 Sample Description	43
5.3.1 Sample Dimensions	43
5.3.2 Sampling Method	43
5.3.3 Sample Preparation	43
5.3.4 Sample Disposition	44
5.4 Test Equipment	44
5.4.1 Equipment Description	44

5.4.2 Coupon Rack	45
5.4.3 Autoclaves	46
5.5 Procedures.....	46
5.5.1 Pre-Test Procedures	46
5.5.2 Test Procedures	47
5.5.3 Post-Test Procedures	47
5.5.4 Data Records	47
5.5.5 Witnesses	48
5.6 Data Analysis	48
5.6.1 Data Reduction Method	48
5.6.2 Data Reported	48
6.0 ANALYSIS OF CHEMICAL COMPOSITION	50
6.1 Applicable Standards and Specifications	52
6.2 General Description of Method	52
6.2.1 Wet Chemical Methods	52
6.2.2 Spectroscopic Methods	53
6.3 Sample Description	53
6.3.1 Sample Dimensions	53
6.3.2 Sampling Method	53
6.3.3 Sample Preparation	54
6.3.4 Sample Disposition	54
6.4 Test Equipment	54
6.4.1 Equipment Description	54
6.4.1.1 Photoelectric Photometer	54
6.4.1.2 Emission Spectroscope	55
6.4.1.3 X-Ray Fluorescence Spectroscope	55
6.5 Test Procedures.....	55
6.5.1 Pre-Test Procedures	55
6.5.2 Procedures	55
6.5.3 Post-Test Procedures	58
6.5.4 Data Records	59
6.6 Data Analysis	59
6.6.1 Data Reduction	59
6.6.2 Data Reported	59
7.0 GRAIN STRUCTURE EVALUATION	61

7.1 Applicable Standards and Specifications	61
7.2 General Description of Method	61
7.3 Specimen Description	63
7.3.1 Sample Method	63
7.3.2 Preparation of Metallographic Specimens	64
7.3.3 Preparation of X-Ray Diffraction Specimens	64
7.3.4 Sample Disposition	65
7.4 Test Equipment	65
7.4.1 Equipment Description for Grain Size	65
7.4.2 Equipment for X-Ray Diffraction Analysis	65
7.5 Test Procedures	65
7.5.1 Grain Size Measurements	66
7.5.2 X-Ray Diffraction analysis	67
7.5.4 Data Records	69
7.5.5 Witnesses	69
7.6 Data Analysis	69
7.6.1 Data Reduction Methodology	69
7.6.1.1 Metallographic Analysis	69
7.6.1.2 X-Ray Diffraction Analysis	69
7.6.2 Data Reported	70

ZIRCONIUM PRESSURE TUBE TESTING
PRODUCTION ASSURANCE PROGRAM
TEST PROCEDURES

1.0 INTRODUCTION

UNC Nuclear Industries (UNC) has initiated a plan for the fabrication of zirconium alloy pressure tubes required for the future operation of N-Reactor. As part of this plan, UNC is establishing a program to qualify and develop a process capable of fabricating these pressure tubes to the requirements of UNC specification HWS 6502, REV 4, Amendment 1. The objective of the Pressure Tube Testing Task is to support the UNC program by performing physical, mechanical and chemical testing on prototype tube sections produced during FY-1986, 1987 and 1988 and to test samples from production runs after 1988 as may be required.

The types of tests included in the Zirconium Pressure Tube Testing Program will be as follows:

- . Tensile tests
- . Burst tests
- . Fracture toughness tests
- . Corrosion tests
- . Chemical composition analyses
- . Grain structure evaluations.

The purpose of this document is to define the procedures that will be used in each type of test included in this task.

2.0 TENSILE TESTING PROCEDURES

The objective of the tensile tests is to determine the strength and ductility of the zirconium tubing material under the conditions of uniaxial tensile stress.

2.1 Applicable Standards and Specifications

Testing will be performed in accordance with the following standards and practices:

ASTM-B353 "Standard Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service"

ASTM-E8, "Standard Methods of Tension Testing of Metallic Materials",

ASTM-E21, "Standard Recommended Practice for Elevated Temperature Tensile Tests of Metallic Materials"

ASTM-E18, "Standard Test Methods for Rockwell Hardness of Metallic Materials and Rockwell Superficial Hardness of Metallic Materials"

ASTM-E4, "Practices of Load Verification of Testing Machines"

ASTM-E83, "Method of Verification and Classification of Extensimeters"

ASTM-E220, "Method for Calibration of Thermocouples by Comparison Techniques"

ASTM-E74, "Practice of Calibration of Force-Measurement Instruments for verifying the Load Indication of Testing Machines"

ASTM-E251, "Test Methods for Performance Characteristics of Bonded Resistance Strain Gages".

2.2 General Description of Method

Tensile tests will be performed on samples taken from the ends of developmental and all prototype tubes and from the ends and central portion of prototype tubes sacrificed for testing. These samples will consist of longitudinal strips of full wall thickness cut from a length of tubing and machined to the form and dimensions of ASTM-E8, Figure 13, Specimen 2.

Prior to testing, the dimensions of the test samples will be measured to the nearest 0.001 inch at the center and both ends of the gage length. The Rockwell hardness of the samples will be taken in the area of the radius between the gage length and the gripper section. This measurement will be according to ASTM-E18. Also, prior to the test program, the tensile testing machine will be verified according to ASTM-E83.

Tensile tests will be performed at room temperature and 300 ± 2 C according to ASTM-E8, ASTM-21 and ASTM-B353. A strain rate of between .003 and .007 in/in.min will be used below and .05 in/in.min above the yield strength. The yield strength will be determined by the 0.2% offset method.

Following testing the specimens will be measured with an accuracy of 0.001 in. to determine the reduction of area at the break using the calculation method recommended in ASTM-E8, Note 22. The results of the tensile and Rockwell hardness tests will be informally reported to UNC within two working days after the test. This report will include 1) yield strength, 2) tensile strength, 3) percentage elongation, and 4) reduction of area. Detailed reports will be submitted at the conclusion of testing.

2.3 Sample Description

2.3.1 Sample Dimensions

The dimensions for the samples will be as recommended in ASTM-E8 and are given in Figures 2 and 3. The ends of the samples will be flattened without heating to accommodate conventional testing machine grips.

2.3.2 Sampling Method

Test samples will be taken from tube lengths approximately 10 in. long supplied to the laboratory by UNC. These tube lengths will be from either short developmental tubes or from full-length production tubes sacrificed for testing. Samples from these sacrificed tubes will be removed from the center and both ends. They may have either as-formed finish or an autoclaved zirconium oxide finish.

2.3.3 Sample Preparation

Test samples will be machined from segments cut from 10 in. lengths of tube as shown in Figure 1. These segments will then be machined to the dimensions and tolerances given in Figures 2 and 3. The end sections of the samples in the region of engagement with the testing machine's grips will be flattened without heating. The gage length will be marked with light punch marks. All machined surfaces will have a surface finish of 63 uin. (as defined in ANSI-B46.1) or better.

All cutting and machining will be performed with the cutting zone generously flooded with a continuously flowing water-based cutting/cooling fluid to prevent heating of the metal. Vise jaws used to hold the tubing and/ samples during cutting and machining will be padded with a soft metal to prevent marring of the surfaces. Following machining, the samples will be permanently identified, and wrapped in sheet plastic and placed in a padded cardboard container for transport

or storage.

Each sample will be identified by a series of characters applied to edge of the gripper section by electric pencil or steel stamp. These characters will include 1) the UNC tube identification number, 2) "TT" to signify that the sample is for tensile testing, 3) the distance from the beginning of the UNC tube to the location of the sample, and 4) the sequence number of the sample prepared for tensile testing. These groups of characters will be separated by hyphens.

2.3.4 Sample Disposition

Following the completion of a test, test samples will be wrapped with sheet plastic and stored in a dry location until the completion of the final report. They will then be transferred to UNC for storage or disposal.

2.4 Test Equipment

2.4.1 Equipment Description

The equipment used in this testing will conform to the requirements specified in ASTM-E8, ASTM-E18 and ASTM-E21. The principal items of equipment include tensile testing machine(s), extensometer(s) and a Rockwell hardness testing machine.

Bare metal thermocouples used in the elevated temperature tests will be formed according to the "Annual Book of ASTM Standards", Vol. 03.04 and calibrated with Bureau of Standards traceability according to ASTM-E220. These thermocouples will be used with standard laboratory digital or strip-chart recorders to continuously record temperature data with an accuracy of ± 3 C.

2.5 Procedures

2.5.1 Pre-Test Procedure

- 1) Prior to the testing of a specimen or a series of specimens, the status of verification of the testing machines, extensometers, and hardness tester and the calibration of thermocouples and associated indicators will be determined. If the verification/calibration of any machine or instrument is not current, the following verification/calibration procedures will be performed, as applicable:
 - . Tensile testing machines will be verified by ASTM-E8 Standard Practices using an elastic-calibration device calibrated in accordance with ASTM-E74 practice.
 - . The Class 2 extensometers will be verified by the ASTM-E83 method.
 - . Rockwell hardness machines will be checked against standardized test blocks according to ASTM-E18 standard test methods, Paragraph 8.1.
 - . Thermocouples will be calibrated according to ASTM-E220 method.
- 2) Before preparation of longitudinal specimens, the sample of tubing from which the specimens are to be machined will be measured at approximately six locations along its length at random diameters; the measurements will be recorded and averaged.
- 3) The specimen(s) will be prepared according to the specifications described in Section 3.0. This includes permanently affixing of an identification number on each sample.

- 4) The test specimens will be visually inspected for smoothness of machined and natural surfaces, and any deviations from specifications will be recorded.
- 5) Measurements will be made of the specimen width and thickness at the center and both ends of the gauge length. These measurements will be recorded to the nearest 0.001 in. in accordance to ASTM-E8.
- 6) A small area on the face of each specimen in the region between the reduced width section that contains the gage length and the full width section engaged by the testing machine grips will be cleaned of all traces of oxide film by mechanical abrasion. The hardness of the specimen in these cleaned areas will be measured on the Rockwell "B" and/or "C" scales using the ASTM-E18 (Section A) Standard Test Method. The measured hardness readings will be recorded to the nearest whole number.

2.5.2 Test Procedure

Room Temperature Tests:

- 1) Mount the sample in the wedge grips of the tensile machine using the precautions described in ASTM-E8, Paragraph 5.2.2; place extensometer on the sample according to the manufacturer's instruction.
- 2) In accordance to ASTM-B353, the specimens will be tested at a strain rate of 0.003 to 0.007 in./in.-min. through the yield strength. After the yield strength has been exceeded, the cross-head speed may be increased to approximately 0.05 in./in.-min. to failure. Autographic or numerical data will be obtained on the stress and corresponding strain during the progress of the test.

Elevated Temperature Tests:

- 1) Mount the specimen onto the pin-mount extensions installed in the grips of the testing machine.
- 2) Mount the extensometer to measure cross-head motion
- 3) Mount the thermocouple bead on the specimen as described in ASTM-E21, Paragraph 9.4.2, and place the temperature control/measurement system into operation.

Mount the furnace on the specimen. The temperature of the specimen will be increased to 300 ± 3 °C in air, taking precautions that the temperature does not overshoot according to ASTM-E21, Paragraph 9.4.6. The time to reach temperature will be noted and recorded.

- 4) In accordance to ASTM-B353, the specimen will be tested at a strain rate of 0.003 to 0.007 in./in.-min. through the yield strength. After the yield strength has been exceeded, the cross-head speed may be increased to approximately 0.05 in./in.-min. to failure. Autographic or numerical data will be obtained of the force applied to the specimen and the corresponding cross-head travel during the progress of the test.

2.5.3 Post-Test Procedures

- 1) The sample will be photographed with emphasis on closeup shots of the region of the bulging and rupture.
- 2) Measurements will be taken of the bursted sample along the circumferential grid lines in the vicinity of the maximum bulge using a profilometer. These measurements will be made with an accuracy of $\pm .002$ in. If the measurements indicate the point of maximum circumference occurs between grid lines, a

measurement will be made at the estimated point of maximum circumference.

- 3) The ruptured metal surfaces will be examined by low power microscope (2-5X) and the fracture will be characterized. Specifically, any evidence of brittle fracture will be noted.
- 4) The sample will be wrapped in plastic and stored in the laboratory unless directed otherwise by UNC. It will be returned to UNC following completion of the final report.

2.5.4 Data Records

All written data will be recorded in laboratory notebooks with numbered pages. Each entry or series of related entries will be dated and initialed by the experimenter. Following submittal of the final report, the notebooks or copies of relevant pages from these notebooks along with all suitably identified stripcharts and/or DAS printouts will be transmitted to UNC for archiving or disposal.

2.5.5 Witnesses

At least one working day prior to any scheduled test described by this test procedure document, the performing organization will notify the UNC program manager of its intent to perform a test. The performing organization will provide access to its facilities for one or more UNC representatives to witness the testing.

2.6 Data Analysis

2.6.1 Data Reduction Methodology

The same data analysis methods will be used in both room temperature and elevated temperature tests.

Yield Strength: The yield strength of the specimens will be determined by the "offset method" described in ASTM-E8 Paragraph 7.3.1.

Tensile Strength: The tensile strength of the specimens will be calculated by dividing the maximum load carried by the specimen during the test by the cross-section area before testing; The cross-section area of longitudinal samples will be calculated by the method described in ASTM-E8 Paragraph 7.1.3.

Elongation: The percentage elongation of the specimen after fractures will be determined by the method of ASTM-E8 Paragraph 7.6.2.

Reduction of Area: The percentage reduction of area of specimens with original rectangular cross-sections will be determined from the original and the final cross-section area determined by method describes in ASTM-E8 Paragraphs 7.7.2 and 7.7.3. For specimens with non-rectangular cross-sections, i.e., the longitudinal samples, only the post-test dimensions at the corners and center of the point of minimum area will be measured and reported.

2.6.2 Data Reported

Preliminary data will be transmitted informally to UNC within two working days following performance of the test. These data will include the following:

- . Specimen number
- . Temperature of test
- . Yield strength
- . Tensile strength
- . Elongation and gage length
- . Reduction of area for specimens with rectangular cross-section; before and after dimensions of the point of minimum area if not rectangular
- . Description of fracture (i.e., cup and cone, brittle, shear)

A comprehensive report will be prepared annually and at the conclusion of this test program that will summarize this information for all specimens considered. It will also include the following additional information:

- . Pre-and Post-test specimen dimensions
- . Strain rate before and after yielding
- . Time to attain test temperature and time at temperature before testing
- . Identification of the equipment used including make and capacity of the testing machine, make and class of extensometer, make and size of furnace, type of temperature controller, and description of thermocouples.
- . Detailed description of testing procedure.

Figure 1 LOCATIONS FROM WHICH LOGITUDINAL TENSION TEST SPECIMENS ARE TO BE CUT.

Dimensions (in.)

G - Gage length	2.000±.005
W - Width (1)	0.500±.010
T - Thickness	Full Wall
R - Fillet radius	1/2
A - Length of reduced section	2-1/4
B - Length of grip section	3 (min.)
C - Width of grip section	11/16 (approx.)

(1) The ends of the reduced section shall differ by not more than .002

Figure 2 DIMENSIONS OF ROOM TEMPERATURE SPECIMENS.

Dimensions (in.)

G - Gage length	2.000±.005
W - Width (1)	0.500±.010
T - Thickness	Full Wall
R - Fillet radius	1/2
A - Length of reduced section	2-1/4
B - Length of grip section	2
C - Width of grip section	2
D - Diameter of pin hole	1/2 (min.)
E - Edge distance from pin	1-1/2 (min.)
F - Hole-fillet distance	1/2
L - Overall length	8 (min.)

(1) The ends of the reduced section shall differ by not more than .002

Figure 3. DIMENSIONS OF SPECIMENS FOR TESTING AT ELEVATED TEMPERATURES.

3.0 BURST TESTING PROCEDURES

The objective of the burst tests is to determine the tensile strength of the zirconium tubing material under biaxial stress conditions and to assure that it is not subject to brittle failure.

3.1 Applicable Standards and Specifications

The burst testing of the zirconium pressure tube samples will be performed in accordance with the following specification:

ASTM-B353-83, Standard Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service, Annex A-1, Recommended Closed-end Burst Testing Procedure for Zirconium Alloy Tubing.

3.2 General Description of Method

Burst tests using water as the pressurizing medium will be performed on zirconium pressure tube samples at room temperature and at 300 C as specified. A typical sample will consist of a short section of pressure tubing with the ends closed by mechanical fixtures clamped to the ends such that the movement of the tubing is not constrained by the closures during testing. The minimum length/diameter ratio of the samples between the end closures will be 10 diameters or approximately 32 inches. Pretest measurements will be made of the outside diameter and wall thickness with an accuracy of ± 0.0002 in.

In the room temperature tests, the sample will be pressurized at a steady rate of 200 psi/min by a hydraulically "stiff" pumping system that does not produce periodic pressure surges with each pump stroke. The system will be instrumented to monitor and record pressure with an accuracy of 1% of the

reading.

In the elevated temperature tests, the samples will be initially pressurized with water to approximately 1000 psi using the same pumping system as used in the room temperature tests. The sample and the water contained in it will be heated to 300 C using either an external furnace or internal sheathed electrical heaters. During heating, water will be bled off to maintain a constant pressure of 1000 psi. After a temperature of 300 ± 2 C is attained, the internal pressure will be increased at a steady rate of 2000 psi/min until bursting is encountered. Following rupture of the sample, it will be removed from the pressurizing apparatus and measurements will be made at room temperature to identify the location of maximum bulge, and the circumferential elongation will be determined at this point, excluding the opening of the rupture, to an accuracy of $\pm .005$ in. Additional measurements will be made to characterize the post-test dimensions of the sample. A small sample of the fracture zone will be removed and metallographically examined to characterize the fracture.

Information that will be reported includes 1) pretest measurements, 2) posttest measurements, 3) maximum fluid pressure, 4) maximum posttest circumferential elongation at the location of rupture, 5) ultimate hoop stress based on the ultimate fluid pressure and pretest sample measurements, 6) the results of the metallographic examination, and 7) the transverse tensile strength as calculated from the test results.

3.3 Sample Description

3.3.1 Sample Dimensions

A test sample will consist of a section of zirconium alloy pressure tubing with 2.70 in. ID and 0.265 in. (nominal) wall thickness. It will be a minimum of 36 inches long which will provide the 10 OD test length required by ASTM B353

(approximately 32in) and sufficient extra length (approximately 4 in) to allow engagement of the end closures.

3.3.2 Sampling Method

Test samples will be taken from tube lengths supplied to the laboratory by UNC and must be cut to length and adequately identified before testing. These tube lengths will be from either short developmental tubes or from full-length production tubes sacrificed for testing. Samples from these sacrificed tubes will be removed from the center and both ends. They may have either as-formed finish or an autoclaved zirconium oxide finish.

3.3.3 Sample Preparation

The samples will be cut to the length specified in Section 3.1 by either reciprocating or band cutoff saw with the cutting zone generously flooded with a continuously flowing water-based cutting/cooling fluid to prevent heating of the metal. Vise jaws used to hold the tubing during cut-off will be padded with a soft metal to prevent marring of the tube surfaces. Following cutting to length, the sample will be permanently identified, wrapped in sheet plastic and placed in a padded rigid wood or metal container for transport or storage.

Each sample will be identified by a series of characters applied to the cut surface by electric pencil or steel stamp. These characters will include 1) the UNC tube identification number, 2) "BT" to signify that the sample has been designated for burst testing, 3) the distance from the beginning of the UNC tube to the face where the identification number is located, and 4) the sequence number of the sample prepared for burst testing. These groups of characters will be separated by hyphens.

For initial planning, it is expected that approximately 20 samples will be burst tested. Approximately 1/2 of these tests will be performed at room temperature and the remainder at elevated temperatures.

3.3.4 Sample Disposition

Following the completion of a test, the test sample will be dried and wrapped with sheet plastic and stored in a dry location until the completion of the final report. It will then be transferred to UNC for storage or disposal.

3.4 Test Equipment

3.4.1 Equipment Description

The equipment used in this burst testing will conform to the requirements specified in ASTM B-353, Annex A1, paragraph A.1.2. This equipment will consist of a test cell, tube closures, pressurization system, heating system, control system and measurement system.

Test Cell

A test cell will be provided to contain the test sample during testing. This test cell will be designed to protect personnel and property from the blast and steam hazards generated by the bursting of the sample at the maximum temperature (300 C) and pressures (<30,000 psi) anticipated. The test sample will be mounted vertically and supported from below so as to not constrain its movement during pressurization. However, both the test assembly and associated equipment will be sufficiently tied down to prevent the generation

of missiles during testing that may damage equipment or be a hazard to personnel.

Tube Closures

Mechanical closures described in HW-76158 "Prototype Burst Test Unit for Irradiated Pressure Tubes", January 1963, will be used to allow pressurizing the samples to failure without imparting axial or radial constraints. These closures are designed to be self-energizing, i.e., the greater the internal pressure the better the seal.

Pressurization System

The pressurization system will consist of a pump, hydraulic accumulator, control valves and regulator valves. It will be capable of smoothly increasing the pressure within the test sample without pump pulsations at the rate of 2000 ± 200 psi/min. Furthermore, it will be capable of maintaining the same volumetric pumping rate established during elastic loading for the remainder of the test, i.e., until bursting is encountered. The pressurization system will be made hydraulically "stiff" as possible by venting all air from the system and using the smallest hydraulic accumulator possible consistent with the requirement that no pump pulsations be present during pressurization.

Heating System

An electric heating system will be provided to heat the sample and the water contained therein to 300 C. A control system will be provided to regulate the temperature of the sample to ± 2 C over the specimen length. The heating

system will consist of either 1) an exterior furnace consisting of a length of pipe to which is affixed electrical strip heaters or 2) internal sheathed electrical heaters. In either case, the heating system will be axially zoned with each zone controlled by its own thermostat to maintain the specified uniformity of temperature. An overtemperature switch will be provided to prevent overheating of the sample. The water entering the sample through the pressurization line will be heated to the temperature of the sample by either routing it through the exterior furnace (if used) or by means of a separate thermostatically controlled electrical preheater.

Control and Regulation System

Valves and controls will be provided to:

- maintain a constant preset fluid volumetric pumping rate.
- regulate the sample temperature to within ± 2 C over the length of the sample.

Measurement System

Instrumentation will be provided to measure and record sample internal pressure and temperatures with accuracies of ± 20 psi and 0.5 C respectively. Temperatures of the sample shall be measured at the top and bottom of the sample as well as at the center and boundary of each heater zone. In no case will fewer than three temperatures be measured and recorded. The indications of sensors will be recorded by either analog or digital instrumentation along with time signals capable of 1 sec resolution. Micrometers with ± 0.0002 in. will be provided for pre- and post test measurements of the test samples. A profilometer with accuracy of $\pm .002$ will be used to take circumferential measurements of the samples before and after testing.

All instrumentation will be calibrated with calibrations traceable to the U.S. Bureau of Standards. Certificates of calibration for all sensors, measurement and read-out instrumentation will be kept on file and made available for inspection by UNC or its agents upon request.

3.5 Procedures

3.5.1 Pre-Test Procedures

The following steps will be performed prior to testing a typical sample:

- 1) The status of the calibration of all instrumentation will be reviewed prior to each test and recalibration of specific sensors or readouts will be performed if certification of Bureau of Standards traceability are not on file.
- 2) The tube section to be tested will be unpacked and inspected for damage such as dents, bending or deep scratches. The results of the inspection will be recorded. If damage is discovered, it will be immediately reported to UNC.
- 3) The tube section will be cut to 36 in. length (+1 in; -0in) and burrs from the cutoff process removed by either a file, machinist's scraper or tapered external/internal reamer. Also, at this time, the identification number will be affixed to the sample using steel stamps or electric pencil, if not already done. See Section 3.0 for numbering method.
- 4) A reference grid will be drawn on the exterior tube surface with aluminum pigment in polymerizing oil using a draftsman's pen while the tube is mounted in a lathe or similar device. This grid will have circumferential lines at 1" increments of length starting at the numbered end and longitudinal lines at 60 degree intervals. One longitudinal line will be identified as a reference for circumferential measurements. The grid will be allowed to dry before the

next step is undertaken.

- 5) Pretest measurements will be made of the sample OD with ± 0.0002 in accuracy at six equally spaced locations around the circumference of the tube at each end and at the center of the sample. The locations will correspond to grid intersections and will be referenced to the distance from the numbered face and the reference longitudinal line. These measurements will be averaged to obtain an average OD measurement. The wall thickness will be measured with ± 0.0002 in accuracy at six equally spaced locations at both ends of the tube. The locations of the measurements will be similarly referenced to the grid. These measurements will be averaged to obtain an average wall thickness; the minimum wall thickness will also be noted.
- 6) The circumference of the tube will be determined with 0.002 in. accuracy using a profilometer at each circumferential grid line. The location of the measurement will be referenced to the distance from the numbered end of the sample.

3.5.2 Test Procedures

- 1) The end fittings will be placed on the end of the sample using a new copper seal ring. This is to be done as follows for each fitting:
 - . fully disassemble end fitting and clean all components;
 - . lubricate all threaded fasteners with graphite-in-oil
 - . assemble jaws into housing
 - . fully retract the jaws by backing out the six pressure screws on the end of the fitting
 - . assemble the Bridgman seal using a new seal ring and mount it to the housing, turning the attachment nut only finger tight
 - . slip the fitting onto the end of the tube
 - . evenly tighten the jaw pressure screws at the end of the fitting

to 40-50 ft-lbs

- tighten the Bridgman seal nut to 75-90 ft-lbs torque

- 2) Mount sample with end fittings applied in test cell.
- 3) Fill sample with water using pressurizing pump; vent air using plug on the ends of the top fitting.
- 4) Pressurize the sample to approximately 100 psi; inspect the sample and pressurizing system for leaks; tighten fittings as required to eliminate leakage.
- 5) Check accumulator for proper loading consistent with manufacturer's recommendations; load as necessary.
- 6) Attach thermocouples for control and measurement of temperature: locations will be as specified in Section 4.0.
- 7) Install furnace (if used); wrap the entire test assembly with 4-6 in of fiberglass insulation or similar material to minimize heat loss (omit this step for room temperature test).
- 8) Pressurize sample to approximately 1000 psi; and hold at this pressure (omit this step for room temperature test).
- 9) Turn on furnace and temperature recording/regulation instrumentation. Uniformly heat the sample to 300 C, taking care to not to exceed this temperature; during heating, pressure in the sample will be maintained constant by the pressure regulating system (omit this step for room temperature test).
- 10) Turn on pressure recording instrumentation. Start and maintain pressurizing

rate of 2000 $\pm$ 200 psi/min by regulating the flow of pressurizing fluid to the sample. When yielding occurs as evidenced by a dropoff of the pressurization rate, continue pressurization by maintaining approximately the same flowrate (e.g., as measured by the stroking rate of the pump or by direct measurement) until bursting occurs. After bursting, the test will be terminated

- 11) The sample will be cooled (if an elevated temperature test), drained, removed from the test stand, and the end fittings removed. Care will be taken not to destroy grid markings on the exterior of the sample.

3.5.3 Post-Test Procedures

- 1) The sample will be photographed with emphasis on closeup shots of the region of the bulging and rupture.
- 2) Measurements will be taken of the bursted sample along the circumferential grid lines in the vicinity of the maximum bulge using a profilometer. These measurements will be made with an accuracy of $\pm .002$ in. If the measurements indicate the point of maximum circumference occurs between grid lines, a measurement will be made at the estimated point of maximum circumference.
- 3) The ruptured metal surfaces will be examined by low power microscope (2-5X) and the fracture will be characterized. Specifically, any evidence of brittle fracture will be noted.
- 4) The sample will be wrapped in plastic and stored in the laboratory unless directed otherwise by UNC. It will be returned to UNC following completion of the final report.

3.5.4 Data Records

All written data will be recorded in laboratory notebooks with numbered pages. Each entry or series of related entries will be dated and initialed by the experimenter. Following submittal of the final report, the notebooks or copies of relevant pages from these notebooks along with all suitably identified stripcharts and/or DAS printouts will be transmitted to UNC for archiving or disposal.

3.5.5 Witnesses

At least 36 hours prior to any scheduled burst test described by the test procedure document, the performing organization will notify the UNC program manager of its intent to perform a test. The performing organization will provide access to its facilities for one or more UNC representatives to witness the testing.

3.6 Data Analysis

3.6.1 Data Reduction Methodology

The post-test circumferential elongation will be determined by subtracting the initial external circumference of the sample from the maximum posttest circumference with both measurements taken at the point of maximum bulge. If the location of the point of maximum bulge occurs between grid lines, the initial measurement will be determined by interpolation between measurements taken at the adjacent grid lines. This elongation will be reported as the percentage of the pretest measurement.

The ultimate hoop strength will be calculated as follows:

$$s = \frac{Pd}{2t}$$

where s = ultimate hoop strength, psi
 p = maximum fluid pressure, psi
 d = average outside diameter minus average wall thickness, in
 t = minimum pretest wall thickness, in.

The ultimate maximum hoop stress will also be evaluated using Lamé's equation as follows:

$$s = p \frac{(D^2 + d^2)}{(D^2 - d^2)}$$

where D = average outside diameter, in.

3.6.2 Data Reported

The following data will be reported:

- . Sample number
- . Controlled temperature of sample
- . Average sample measurements
- . Minimum wall thickness
- . Maximum fluid pressure
- . Ultimate hoop stress calculated by the equations given in Section 6.1
- . Total circumferential elongation

Preliminary data will be transmitted informally to UNC within two working days following performance of the test. A final report will be prepared that will

include this information as well as complete tabulations of sample measurements, and the pressure histories and temperature histories recorded during the test.

4.0 FRACTURE TOUGHNESS TESTING

The objective of the fracture toughness tests is to determine the plane-strain fracture toughness of zirconium tubing produced during UNC's manufacturing development program and subsequent prototype runs. Specifically, these tests will be directed at determining the property K_{IC} . This property characterizes the resistance of the tube material to fracture in the presence of a sharp crack under severe tensile constraint and is believed to represent a lower limiting value for fracture toughness. This value may be used to estimate the relation between failure stress and defect size for the material in service.

4.1 Applicable Standards and Specifications

Testing will be performed in accordance with the following standards and practices:

ASTM-E399-83, "Standard Test Method for Plane Strain Fracture Toughness of Metallic Materials".

ASTM-E813-81, "Standard Test Method for J_{IC} A Measure of Fracture Toughness".

ASTM-E616-82, "Standard Terminology Relating to Fracture Testing".

ASTM-E4-83a, "Practices for Load Verification of Testing Machines".

4.2 General Description of Method

Tests will be performed at room temperature and at 300 C to determine K_{IC} for compact specimens machined from short lengths of zirconium tubing removed from the ends and/or center sections of developmental, prototype and production tubes. These specimens will be the standard ASTM-E399 0.25 in. specimen with 0.20 minimum

thickness shown in Figure 4.1. Specimens will all be oriented in the C-L direction (see ASTM-E616 for terminology). Prior to testing, the specimens will be fatigue precracked by cyclically loading according to Annex 2 of ASTM-E399.

The size of the sample described above is essentially the maximum that is obtainable by considering the configuration and dimensions of the tubing. Before testing, it may not be possible to determine whether ASTM-E399 or ASTM-E813 will apply for the range of toughness that will be encountered. Therefore, a procedure will be used that will be responsive to both standard test methods.

The procedure that will be used involves the loading of the precracked specimen at the rate of 30,000 to 150,000 psi in.^{1/2}/min while autographically recording load and displacement of the mouth of the specimen notch according to ASTM-E399. Concurrently, the crack progress will be recorded autographically using the electropotential method responsive to ASTM-E813, Paragraph 8.3.4. Therefore, data will be obtained that can be analyzed by methods prescribed in both standards.

The test data will be first analyzed by methods prescribed by ASTM-E399 to determine K_{IC} and the results will be tested for validity. If invalid, J_{IC} will be determined according to ASTM-E813 and K_{IC} will be estimated from it. If ASTM-E813 is also found invalid, it can be concluded that the material is very tough, and other methods must be investigated to quantify toughness, e.g., the use of the specimen strength ratio (R_{SX}) and plastic work (area under the load vs. head travel curve).

Following data analysis, the preliminary results will be transmitted informally to UNC within 5 working days. These results will include: 1) specimen dimensions, 2) test temperature, 3) crack plane orientation, 4) fracture surface appearance, 5) K_{IC} , and 6) applicable ASTM Standard. Comprehensive reports will be prepared at the end of each fiscal year's activities and at the conclusion of the program.

4.3 Sample Description

4.3.1 Specimen Dimensions

Test specimens will be of the configuration and dimensions of the "compact specimen" described in ASTM-E399 and shown in Figure 4.1. These specimens will be machined from the wall of a tube section and will be oriented such that the crack plane is in the C-L direction as defined in ASTM-E616. A crack starter notch will be provided with an integral knife-edge, to allow the use of a standard "clip-in" displacement gage.

4.3.2 Sampling Method

Test specimens will be taken from tube lengths supplied to the laboratory by UNC and must be machined and adequately identified before testing. A minimum 2-1/2-in. length will be required. These tube lengths will be from either short developmental tubes or from full-length prototype tubes sacrificed for testing. Specimens from these sacrificed tubes will be removed from the center and both ends. They may have either as-formed finish or a specially oxidized finish.

4.3.3 Specimen Preparation

The longitudinal specimens for testing at room temperature will be machined from segments cut from 2-1/2-in. (minimum) lengths of tube as shown in Figure 4.2. These segments will then be machined to the dimensions and tolerances given in Figure 4.1. The straight-through notch design will be used as detailed in Figure 4.3. The crack starter notch will also be provided with an integral knife edge as shown in Figure 4.4.

For the mounting of the specimens in the testing machines, a pair of clevises will be machined from a high yield strength (280 Ksi) steel according to Figure

4.5.

All cutting and machining will be performed with the cutting zone generously flooded with a continuously flowing water-based cutting/cooling fluid to prevent heating of the metal. Vise jaws used to hold the tubing and/samples during cutting and machining will be padded with a soft metal to prevent marring of the surfaces. Following machining, the samples will be permanently identified, and wrapped in sheet plastic and placed in a padded cardboard container for transport or storage.

Each sample will be identified by a series of characters by electric pencil applied to the edge or on the face away from the crack zone. These characters will include 1) the UNC tube identification number, 2) "TT" to signify that the sample is for toughness testing, 3) the distance from the beginning of the UNC tube to the location of the sample, and 4) the sequence number of the sample prepared for testing. These groups of characters will be separated by hyphens.

4.3.4 Sample Disposition

Following the completion of a test, test samples will be wrapped with sheet plastic and stored in a dry location until the completion of the final report. It will then be transferred to UNC for storage or disposal.

4.4 Test Equipment

4.1 Equipment Description

The equipment used in this testing will conform to the requirements specified in ASTM-E399 and ASTM-E813. The principal items of equipment include 1) tensile testing machine(s) with digital or analog autographic load and strain recorders, 2) furnace (with controls), 3) clip-on crack displacement gage(s), and 4) electropotential crack growth monitor. An electropotential crack monitor will be

fabricated and calibrated as described in UNI 3602, "Post Irradiation Examination of Pressure Tubes 2755 and 1054 Pt. 2: Fracture Toughness and Tensile Tests", (Nov. 1985).

Base metal thermocouples will be formed according to "Annual Book of ASTM Standards", Vol. 03.04 and calibrated with Bureau of Standards Traceability according to ASTM-E220. These thermocouples will be used with standard laboratory digital or strip-chart recorders to continuously record temperature data with an accuracy of ± 3 C.

4.5 Test Procedures

4.5.1 Pre-Test Procedures

- 1) Prior to the testing of a specimen or a series of specimens, the status of verification of the testing machines and crack displacement gages and the calibration of thermocouples with associated indicators will be determined. If the verification/calibration of any machine or instrument is not current, the following verification/calibration procedures will be performed, as applicable:

- . Tensile testing machines will be verified by ASTM-E8 Standard Practices using an elastic-calibration device calibrated in accordance with ASTM-E74 practice.
- . The crack displacement gage will be calibrated according to ASTM-E399, Paragraph 6.3.2.
- . The electropotential crack growth monitor shall be calibrated according to standard laboratory procedures.
- . Thermocouples will be calibrated according to the ASTM-E220 method.

- 2) A fatigue precrack will be produced at the end of the specimen's crack starter notch by cyclically loading the specimen with a tensile testing machine according to the method prescribed in ASTM-E399, Annex A2. This loading will be cycled with a ratio of minimum to maximum stress between -1 and +0.1 until a crack is produced that is $0.50W$ long (the length of the crack is defined as the length of the starter notch plus the fatigue crack; W is $1/2$ of the thickness of the specimen). Precracking will be performed at room temperature; however, when specimens for testing at elevated temperatures are precracked, the additional provisions specified in ASTM-E399, Annex A2, Section A2.4.4 will be followed.
- 3) The following measurements will be made:
 - . The width W to the nearest 0.001 in. at not less than three positions near the notch location and record the average value
 - . The crack length to the nearest 0.5% of the overall dimension as described in ASTM-E399, Paragraph 8.2.2.

4.5.2 Test Procedures

- 1) The clevises and their attachments will be mounted to the testing machine. The upper and lower rod centerlines will be checked for alignment within 0.03 in. as required by ASTM-E399, Paragraph A4.4.2.
- 2) The precracked specimen will be mounted in the clevises so that the center of the specimen mounting pins are within 0.03 in. of the center of the clevis openings.
- 3) The clip-on crack extension gage and the leads for the electropotential crack extension instrumentation will be mounted on the specimen.

- 4) For the heated tests, the thermocouple will be attached to the specimen and the furnace will be installed. The specimen will be heated in air to 300 C taking care to not overshoot this temperature; this temperature will be held for at least 20 minutes. The time required to heat the specimen and the holding time before the test commences will be recorded.
- 5) The specimen will be loaded at such a rate that the rate of increase of stress intensity is between 30 to 150 Ksi in^{1/2}/min. according to ASTM-E399, Paragraph A4.4.2.1. Load, output of the extension gage and the output of the electropotential crack extension gage will be recorded autographically.
- 6) The test will be continued until the specimen can no longer support the load; the maximum load (P_{max}) will be recorded.

4.5.3 Post-Test Procedures

- 1) The ruptured metal surfaces will be examined visually and the fracture will be characterized by type as illustrated in ASTM-E399, Figure 9.

4.5.4 Data Records

All written data will be recorded in laboratory notebooks with numbered pages. Each entry or series of related entries will be dated and initialed by the experimenter. Following submittal of the final report, the notebooks or copies of relevant pages from these notebooks along with all suitably identified stripcharts and/or DAS printouts will be transmitted to UNC for archiving or disposal.

4.5.5 Witnesses

At least one working day prior to any scheduled test described by this test procedure document, the performing organization will notify the UNC program

manager of its intent to perform a test on a precracked specimen. The performing organization will provide access to its facilities for one or more UNC representatives to witness the testing. UNC may choose to exercise or waive its right to witness any or all tests.

4.6 Data Analysis

4.6.1 Data Reduction Methodology

- 1) The data will be first analyzed by the method prescribed in ASTM-E399 (paragraph numbers in the following list refer to this standard):
 - . The load P_Q will be calculated from the autographic recording of load vs displacement according to Paragraph 9.9.1.
 - . Using this value of P_Q along with the pre-test measurements of specimen thickness (B) and crack length (a), K_Q will be calculated by equations given in Paragraph A4.5.3.
 - . The specimen strength ratio will be calculated using the equation given in Paragraph A4.5.4.
 - . The validity of the test results will be tested using criteria specified in Paragraphs 9.1.2 and 9.1.3, i.e., $P_{max}/P_Q < 1.10$ and $\{2.5(K_Q/s_{ys})\} < B$ (where s_{ys} is the 0.2% offset yield strength in tension). If true, then $K_Q = K_{IC}$.
- 2) If the method of data analysis recommended by ASTM-E399 is shown to be invalid in Step 2 above, then the method of ASTM-E813 for single samples will be used to estimate K_{IC} (paragraph numbers in the following list refer to this standard):

FIGURE 1 Compact Specimen Proportions and Dimensions: $B = 0.250\text{-in.}$ nominal (0.200-in. minimum).

FIGURE 2 Location of Specimen Removal.

- Fatigue crack extension on each surface of the specimen containing a straight-through notch shall be at least $0.025 W$
- Crack starter notch shall be perpendicular to the specimen surfaces and to the intended direction of crack propagation within $\pm 2^\circ$.

FIGURE 3 Details of Crack Starter Notch.

- NOTE 1—Dimensions in inches.
- NOTE 2—Gage length shown corresponds to clip gage spacer block dimensions shown in Annex A1, but other gage lengths may be used provided they are appropriate to the specimen (see 6.3.3).

Metric Equivalents				
in.	0.050	0.060	0.200	0.250
mm	1.3	1.5	5.1	6.4

FIGURE 4 Details of Integral Knife Edge for Clip-on Gage.

A—Surfaces must be flat, in-line, and perpendicular as applicable, to within 0.05 mm.

NOTE 1—Pin diameter = $0.24W + 0.000, - 0.005W$.

NOTE 2—Flat-bottom hole is a modified E 399 design.

FIGURE 5 Clevis for Mounting Specimen in Testing Machine.

5.0 CORROSION RATE TESTING

The purpose of corrosion rate testing is to provide a high degree of confidence that the production tubes will have adequate corrosion resistance for long term service in N-Reactor.

5.1 Applicable Standards and Specifications

Corrosion rate testing will be performed in accordance with the following standards and practices:

ASTM-B353, "Standard Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service".

ASTM-G-15, "Standard Definitions of Terms Relating to Corrosion and Corrosion Testing".

ASTM-G-31, "Standard Recommended Practice for Laboratory Immersion Corrosion Testing of Metals".

ASTM-G-1, "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens".

ASTM-G-46, "Standard Recommended Practice for Examination and Evaluation of Pitting Corrosion".

ASTM-G-16, "Standard Recommended Practice for Applying Statistics to Analysis of Corrosion Data".

HWS-6502, Rev. 4, Amendment 1, June 20, 1985, "Specification for

Zircaloy-2 Pressure Tubes for 105-N, Project H-700".

ASTM-G-2, "Standard Practice for Aqueous Corrosion Testing of Samples of Zirconium and Zirconium Alloys".

ASTM-E220, "Method for Calibration of Thermocouples by the comparison method".

5.2 General Description of Method

Thin corrosion test coupons will be cut from the excess length at the end of production tubes and from several locations along the length of sacrificial tubes produced during UNC's manufacturing development program. The cut surfaces will be smoothed and the coupons will be cleaned, degreased, and then etched to remove 0.001 to 0.002 inch of metal from all surfaces. After rinsing and drying, each coupon will be examined for scratches and other surface imperfections under a low power microscope. Coupons having excessive scratches or surface imperfection which render them unusable for corrosion testing will be discarded. Usable coupons will be measured for size. Each coupon will be weighed twice to the nearest 0.05 milligrams using different scales. The average of the two weights (if they agree within 0.2 milligram) will be used for weight gain (or loss) calculations. The coupons will then be placed on racks so that they do not touch each other, and the racks placed in an autoclave with deoxygenated, demineralized water. The corrosion test will be performed on all development tubes at 750°F (399°C) and 1500 psi (10.3 MPa) for 3 days. Similar tests will be performed on the first two prototype tubes. Average weight gain of the corrosion coupons shall not be more than 22 mg/dm² in a 72 hour test or 38 mg/dm² in a 336 hour (14 days) test.

5.3 Sample Description

5.3.1 Sample Dimensions

Corrosion testing coupons should have a high surface area to volume so that small weight changes may be accurately measured. Such coupons may be machined from rings cut from the tubes as described in the next paragraph.

5.3.2 Sampling Method

Two-inch long rings will be cut from the tubes to be samples, either from the ends or any other point to be sampled. Material will be machined from the OD of the tube to make an annular ring 1/16 in. thick. These rings will be cut into specimens 1 in. long and 2" circumferential dimension. All cutting and machining will be performed with adequate cooling to prevent heating of the specimens.

5.3.3 Specimen Preparation

Each coupon to be corrosion tested will be prepared as follows:

1. Degrease in methyl or ethyl alcohol. Ultrasonic cleaning may be used.
2. If necessary abrade all edges and broad surfaces with extra fine abrasive paper to the point where no pits or other superficial damage such as scratches are visible.
3. Rinse thoroughly with deionized water and dry with alcohol.
4. Identify each coupon with a number or symbol scribed on one end of a broad surface with a vibratory pencil.
5. Measure each dimension to the nearest 0.03 millimeter and weigh each coupon to

+0.05 milligram. Dimensions and weights will be checked with an independent instrument.

5.3.4 Sample Disposition

Following the completion of a corrosion test, test coupons will be placed in labeled envelopes and stored in a dry location (such as desicator) until the completion of the final report. Coupons will then be transferred to UNC for storage or disposal.

5.4.0 Test Equipment

5.4.1 Equipment Description

The equipment used in this testing will conform to the requirements specified in ASTM-G-2. The principle items of equipment include (1) micrometer, (2) analytical balance, (3) coupon rack, and (4) autoclave(s). Instruments will conform to ASTM standards and where necessary will be calibrated with Bureau of Standards traceability according to ASTM-E220. The autoclave control system shall be adequate to control temperature to $\pm 3^{\circ}\text{C}$ and pressure to ± 100 psi. A strip chart recorder will continuously record autoclave temperatures.

Micrometer

A micrometer will be used which is capable of measuring to ± 0.03 millimeter.

Analytical Balance

Balances will be used that are capable of weighing to ± 0.05 milligram.

5.4.2 Coupon Rack

The rack for holding coupons will be made of 300 series stainless steel. It

will be capable of keeping the coupons from touching each other during testing. Contact surface area between coupon and holder will be minimized.

5.4.3 Autoclave(s)

Existing autoclave(s) constructed from 300 series stainless steel and manufactured to conform with ASME and state regulations governing unfired pressure vessels will be used. Autoclaves shall be fitted with devices for measuring temperature and pressure, safety devices, and venting valves.

5.5 Test Procedures

5.5.1 Pre-Test Procedures

Prior to testing coupons, the calibration status of all instruments and devices will be verified. Any out-of-date or unverifiable calibration will be recalibrated.

The autoclave(s) and coupon rack(s) accordingly will be prepared to ASTM G-2, Section 11.

Coupons prepared according to Section 3.0 will be weighed and properly mounted on the coupon rack(s). Loaded rack(s) will then be mounted into the autoclave(s).

The autoclave(s) will be filled to the appropriate level with deionized and degassed water with conductivity less than 2 micromhos. Seal the autoclave(s) correctly.

5.5.2 Test Procedures

When the autoclave(s) has been properly sealed and checked, the heating

elements will be activated. The system will be brought to test temperature of $399^{\circ}\text{C} \pm 3^{\circ}\text{C}$. This temperature will be maintained for 72 hours. After 72 hours at temperature, the power source will be deactivated.

5.5.3 Post-Test Procedures

When the system has cooled to room temperature, the autoclave will be opened and the coupons will be removed from the rack(s) using clean gloves and/or plastic-tipped tweezers. Rinse and dry the coupons and store them in a clean, dry container while they equilibrate with the balance for at least one hour. (Coupons and balance must be at the same temperature to prevent convection drafts in the balance enclosure.)

Weigh the coupons; report corrosion results as the weight change (mg) per unit area (dm^2). Report also the color, luster, and surface irregularities of each coupon. The final report will include color photographs of typical specimens and any unusual findings.

Water remaining in the autoclave after testing will be sampled to determine its pH and conductivity.

5.5.4 Data Records

All written data will be recorded in laboratory notebooks with numbered pages. Each entry or series of related entries will be dated and initialed by the experimenter. Following submittal of the final report, the notebooks or copies of relevant pages from these notebooks along with all suitably identified stripcharts and/or DAS printouts will be transmitted to UNC for archiving or disposal.

5.5.5 Witnesses

At least one working day prior to any scheduled test described by this test procedure document, the performing organization will notify the UNC program manager of its intent to perform a test. The performing organization will provide access to its facilities for one or more UNC representatives to witness the testing.

5.6 Data Analysis

5.6.1 Data Reduction Method

Using measured weights from before and after testing, and coupon surface area from pretest measurements, the corrosion rates will be calculated and expressed as weight change (mg) per unit area (dm^2). An error analysis will be performed to determine the uncertainty in the results.

5.6.2 Data Reported

Preliminary data will be transmitted informally to UNC within two working days following performance of a test. These data will include:

- . tube number
- . coupon numbers
- . test temperature
- . corrosion rates (mg/dm^2)
- . color of oxide
- . luster of oxide
- . surface irregularities, if any.

A comprehensive report will be prepared at the end of each fiscal year's effort and at the conclusion of this test program that will summarize this

information for all tube specimens tested.

The final report will also include the following additional information:

- . coupon dimensions
- . coupon description (scratches, etc.)
- . pre- and post-test coupon weights
- . time to attain testing temperature
- . time at temperature
- . equipment identifications and descriptions
- . detailed testing procedure.

6.0 ANALYSIS OF CHEMICAL COMPOSITION

A nuclear grade zirconium alloy (very similar to UNS R60802, which has been qualified for N-Reactor service) is specified as the raw material for the pressure tubes. The specification identifies five elements that must be within controlled limits, and 19 impurities that must be less than specified limits. Table 1 shows this required composition. The objective of the chemical composition analyses is to determine the compliance of the pressure tubes with this specification after fabrication. It is anticipated that analyses for all elements will be performed on all ingots by the vendor. Tubing samples during production runs need only be analyzed for hydrogen, oxygen and nitrogen. However, during fabrication process development, it is considered necessary to perform complete analyses on a sufficient number of tubing samples to assure correspondence with ingot compositions.

Methods that will be used in these analyses include wet chemical, spectrophotometric, emission spectrometric, and x-ray fluorescent methods. Specific methods described are typical. If the performing laboratory prefers alternative methods of equal or better accuracies to those described, these methods will be submitted to UNC for approval prior to the start of testing.

TABLE 1. Chemical Composition of Zirconium Alloy Ingots
and Pressure Tubes for N-Reactor Service*

<u>Chemical Requirements</u>		<u>Zircaloy-2</u>	
Tin		1.20% - 1.70%	
Iron		0.07% - 0.20%	
Chromium		0.05% - 0.15%	
Nickel		0.03% - 0.08%	
Impurities		See below	
Zirconium		Remainder	
Iron & Chromium & Nickel		0.18 to 0.38	

<u>Impurities</u>			
<u>Element</u>	<u>Maximum ppm</u>	<u>Element</u>	<u>Maximum ppm</u>
Aluminum	75.0	Manganese	50.0
Boron	0.5	Molybdenum	50.0
Cadmium	0.5	Nitrogen	80.0
*Carbon	320 (200 minimum)	*Oxygen	1000.0-1500.0
*Cobalt	10.0	*Silicon	250.0 (150 minimum)
Copper	50.0	*Sodium	20.0
Hafnium	100.0	Titanium	50.0
Hydrogen	25.0	Tungsten	100.0
*Lead	100.0	Uranium	3.5
Magnesium	20.0	*Vanadium	50.0

Tubing produced to this specification will be fabricated from reactor grade Zirconium sponge with up to 50% scrap and shall conform to Zirconium Alloy Grade ASTM UNS Number R60802 (with exceptions marked by asterisk in the table).

6.1 Applicable Standards and Specifications

Analytical chemistry testing will be performed in accordance with the following standards and practices:

- 1) HWS 6502, Rev. 4, Amendment 1, June 20, 1985, "Specification for Zircaloy-2 Pressure Tubes for 105-N, Project H-700".
- 2) ASTM-B353-83, "Standard Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Services," 1983.
- 3) ASTM-E146-83, "Standard Methods for Chemical Analysis of Zirconium and Zirconium Alloys," 1983.
- 4) ASTM-E-572-81, "Standard Method for X-Ray Emission Spectrometric Analysis of Stainless Steel," 1981.
- 5) UNC Analytical Procedures for Zirconium and Zirconium Alloys: EC-2, EI-1, EN-1, EN-3, and ET-1. (Based on ASTM E146.)

6.2 General Description of the Methods

6.2.1 Wet Chemical Methods

Wet methods, including spectrophotometric methods, are available for almost all of the elements specified. These methods require dissolution of the samples, which takes extra time and often a certain amount of extra care and skill in transferring and processing the solutions. For low atomic number elements, however, there is no alternative. Hydrogen, boron, carbon, nitrogen and oxygen are not visible in emission or X-ray spectra, so they are measured with spectrophotometric, volumetric, extraction or combustion methods.

6.2.2 Spectroscopic Methods

Spectroscopic methods, when they can be used, are convenient because they can be performed with solid samples taken directly from the specimen to be analyzed. In these analyses, either emission spectroscopy or X-ray fluorescence will be used depending on its applicability and accuracy on the elements to be analyzed. In emission spectroscopy the sample is excited by sparking it, while in X-ray fluorescence it is excited by X-rays. Both excitation methods produce spectra that can be calibrated and analyzed for characteristic frequencies of the elements present in the specimen.

6.3 Sample Description

6.3.1 Sample Dimensions

No special geometric size or dimension is required for chemical analysis samples. The important characteristics are that samples should be sufficiently large to be chemically uniform and representative of the tube being analyzed. Sufficient sample should be taken to permit re-analysis, if necessary, and for ultimate transmittal to UNC. Some methods may require that a known weight of sample be used. Weighing should be done on an analytical balance capable of weighing to ± 0.0001 gram.

6.3.2 Sampling Method

In general, turnings, millings, or drillings make satisfactory samples for chemical analyses. They should be taken deeply enough in the metal that they are representative of the bulk material. For the determination of hydrogen, nitrogen and oxygen, solid pieces are preferred in order to minimize the surface area of the sample. All samples should be cut in a manner that avoids heating and contamination by cutting fluids and lubricants.

6.3.3 Sample Preparation

Prior to use, samples should be stored in glass vials with plastic lids. Each sample should be washed twice in deionized water and then dried by rinsing them twice in clean acetone, air drying and storing them. Samples for carbon analysis should not contact paper or waxed cardboard as in screw-capped lids.

6.3.4 Sample Disposition

Upon completion of the analysis, remaining sample should be stored in its vial in a dry location until the final report is completed. Samples will then be transferred to UNC for storage or disposal.

6.4 Test Equipment

6.4.1 Equipment Description

The equipment used in this testing will conform to the requirements specified in ASTM E146 and other referenced descriptions of the applicable analytical procedures. Many standard laboratory devices are required, e.g., analytical balances, pH mmeters, furnaces, volumetric glassware, etc. In addition, a few specialized items are listed below. Equipment must be in properly calibrated condition for use during the pressure tube fabrication program.

6.4.1.1 Photoelectric Photometer

This is a standard instrument found in any well-equipped analytical laboratory. It must provide a monochromatized radiant energy source, absorption cells and an arrangement for photoelectric measurements. The choice of a specific instrument will be a function of price, need for special accuracy or features, and personal preference of the analyst.

6.4.1.2 Emission Spectroscope

This, also, is a standard laboratory instrument, albeit somewhat more sophisticated than the basic photometer. It must have an excitation source, usually a graphite counter electrode, a spectrometer with sufficient resolving power, and an adequate photometric measuring system. Again, there are several adequate instruments, and the one to be utilized will depend on certain technical and subjective factors.

6.4.1.3 X-Ray Fluorescence Spectroscope

This is a somewhat less common, but nevertheless standard analytical instrument. It consists of a source capable of producing specific mono-energetic X-rays, a mechanism for irradiating the target, and appropriate detectors for collecting and measuring the fluorescent radiation from the target.

6.5 Test Procedures

6.5.1 Pre-Test Procedures

Prior to analyzing a sample or a series of samples, the calibration and operating condition of the instrumentation will be verified. Reference samples may be analyzed to check the status. If the verification or calibration of an instrument is not current, it will be made so by proper calibration procedures carried out by qualified personnel. Analysts performing the analyses will also be properly qualified for the instruments being used.

The samples will be taken and prepared in accordance with Section 3.0.

6.5.2 Procedures

Complete detailed procedures are given in the referenced documents. The

following is a list of recommended methods for the named elements. Proposed alternative methods giving comparable or better accuracies than these recommended methods should be submitted to UNC for approval.

Tin by the Iodate Titration (Volumetric) Method

The tin, in solution, is reduced to the stannous form with aluminum in a hydrochloric-hydrofluoric acid medium. Stannous tin is titrated in a carbon dioxide atmosphere with standard iodate solution, using starch as the indicator.

Iron by the Ortho-Phenanthroline (Photometric) Method

Ferrous iron, in a solution having a pH of 5.5 to 6.0, forms an orange-red complex with o-phenanthroline. Photometric measurement is made at 508 nanometers (nm).

Chromium by the Diphenylcarbazide (Photometric) Method

Chromium, in the hexavalent state, forms a red-violet complex with diphenylcarbazide in a sulfuric acid solution. Photometric measurement is made at approximately 540 nm.

Nickel by the Dimethylglyoxime (Photometric) Method

Nickel forms a red-color, soluble nickelic salt with dimethylglyoxime. Photometric measurement is made at approximately 540 nm.

Hydrogen by the Hot Extraction Method

Solid samples, contained in a molybdenum crucible in a vacuum system, are subjected to induction heating to remove hydrogen. The hydrogen, which is the only gas evolved at the temperature used, is transferred from the heating zone

with a high-speed mercury diffusion pump to a suitable calibrated volume. The pressure in the volume is measured with a McLeod gage to determine the amount of hydrogen present.

Boron

Method to be provided by performing laboratory.

Carbon by the Combustion-Conductometric Method

The sample is burned in a stream of oxygen, and the carbon dioxide in the evolved gases is absorbed by a barium hydroxide or sodium hydroxide solution. The amount of carbon present in the sample is determined by measuring the change in conductance of the absorbing solution.

Nitrogen by the Nessler (Photometric) Method

The sample is dissolved in hydrochloric-hydrofluoric acid solution, which is then made basic. Nitrogen is separated as ammonia by steam distillation. Nessler's reagent is added to the distillate to form the yellow ammonium complex. Photometric measurement is made at approximately 430 nm.

Oxygen by the Inert Gas Fusion Method Using the Platinum Flux Noncycling Technique

This method is based on the fusion of sample and flux in a graphite crucible under a flowing argon atmosphere. The oxygen in the sample reacts with the carbon to form carbon monoxide. The effluent gases are swept through iodine pentoxide to quantitatively convert the carbon monoxide to carbon dioxide. The free iodine formed in this reaction is removed by sodium thiosulfate. The gas mixture is dispersed into a solution of barium hydroxide or sodium hydroxide where the carbon dioxide forms barium carbonate or sodium carbonate. The resulting change in the

resistance of the solution is a measure of the oxygen content of the sample.

Impurities by Emission Spectroscopy

A solid sample of the specimen, which may be turnings or drillings, is excited by sparking it with a graphite electrode. Electrons in each element are raised to higher energy levels. As they fall back to their normal states light is emitted at frequencies characteristic of each element. By measuring the spectrum (intensity as a function of frequency) the elements present can be identified and quantified against a calibration spectrum.

Impurities by X-Ray Fluorescence

A solid sample, as in 5.2.10, is excited with mono-energetic X-rays. The sample will fluoresce, producing a spectrum similar to the emission spectrum, except that the emitted photons are at much higher energies, in the X-ray region of the electromagnetic spectrum. By counting the emitted X-rays at frequencies that are characteristic of each element, the presence and concentration of the elements may be determined from a calibration curve. Elements in the periodic table from aluminum through cerium are analyzed through their characteristic K X-ray lines. Tungsten through uranium are analyzed with the L X-ray lines. Special techniques may be employed when interferences occur at the normally-used frequencies.

6.5.3 Post-Test Procedures

Residual sample material will be stored in well-labelled glass vials. After completion of the final report they will be returned to UNC for archiving or future disposal.

6.5.4 Data Records

All analytical results will be properly identified and recorded in laboratory record books with numbered pages. Each entry or series of related entries will be dated and signed by the analyst or project engineer. Following submittal of the final report, the notebook pages will be transmitted to UNC for archiving or disposal.

6.6.0 Data Analysis

6.6.1 Data Reduction

Using pertinent calibration curves for the instruments utilized, the concentration of each element or group of elements in the material specification (see Table 1 in Section 1.0) will be calculated along with error limits.

6.6.2 Data Reporting

Within two working days following completion of the analysis, the following preliminary data will be reported to the UNC Project Manager or his designated representative:

- a) Tube number.
- b) Date of analysis.
- c) Physical form of sample.
- d) Method of taking sample.
- e) Unusual or off-standard observations.
- f) Concentrations in percent by weight of all of the elements specified with a concentration range. The allowable range should be indicated on the form. These elements are:

<u>Element</u>	<u>Range, %</u>
Tin	1.20 - 1.70
Iron	0.07 - 0.20
Chromium	0.05 - 0.15
Nickel	0.03 - 0.08
Oxygen	0.10 - 0.15
Iron & Chromium & Nickel	0.18 - 0.38

- g) Concentrations in ppm of each of the impurity elements that are specified with a maximum allowable concentration. The allowable concentration shall be indicated on the form.
- h) The presence of any unspecified element at an estimated concentration above 50 ppm.
- i) Any off-standard procedures that was used.

A comprehensive report will be prepared at the end of each fiscal year's activities and at the conclusion of this test program, that will summarize this information for all of the specimens analyzed. These reports will include the above information in a summary format plus descriptions of the instrumentation and procedures used.

7.0 GRAIN STRUCTURE EVALUATION

The objective of this task is to evaluate the grain structure of zirconium alloy pressure tubes for subsequent prediction by UNC of their long term serviceability under in-core conditions. The long term satisfactory performance of zirconium alloy pressure tubes for N-Reactor under service conditions is influenced by grain structure of the metal. Through experience gained in the study of the past service history of N-reactor pressure tubes, the range of desirable grain structure characteristics have been identified and will be used as guidelines for the development of UNC manufacturing processes. These characteristics include 1) grain size in the radial, transverse and longitudinal directions, 2) basal pole orientation (texture), and 3) the orientation of intermetallic precipitates if present. This task is concerned principally with determining these characteristics of selected samples of prototype tubes.

7.1 Applicable Standards and Specifications

Testing will be performed in accordance with the following standards and practices:

ASTM-E-7-73	"Standard Definition of Terms Relating to Metallography"
ASTM-E-3-80	"Standard Methods of Preparation of Metallographic Specimens"
ASTM-E-340-68	"Method for Macroetching Metals and Alloys"
ASTM-E-112-84	"Standard Method for Determining Average Grain Size"
ASTM-E-81-77	"Preparing Quantitative Pole Figures of Metals"
ASTM-E-883-83	"Practice for Metallographic Photomicrography"

7.2 General Description of Method

Metallographic and X-ray diffraction tests will be performed on samples taken from the ends of prototype tubes chosen for evaluation and from the center and

ends of sacrificed prototype tubes. The samples will consist of rings of the full tube wall, about 1/2 inch long for metallography specimens and 2 inches for the X-ray diffraction samples. To produce metallography specimens, a ring will be divided into circumferential segments to form a number of specimens. Three specimens will be used for metallographic examination and the remainder will be adequately identified and preserved for archival purposes.

The metallographic specimens will be prepared according to ASTM-E-3. They will be cleaned and mounted in polyester or epoxy resin. One sample will be mounted to allow examination of the transverse section (orientation "F" as designated in ASTM-E-3). The others will be mounted to examine the radial longitudinal section ("G") and the tangential longitudinal Section ("H"). The specimens will be permanently identified and rough ground, fine ground and polished either by hand or machine. After polishing, they will be cleaned, lightly etched, desmutted (if required) and washed using the solutions and procedures specified in ASTM-E-340. The specimens will then be metallographically examined to determine grain sizes using the procedures of ASTM-E-112. Any intermetallics or voids present will be characterized, and the thickness of the oxide layer determined. Color photomicrographs will be taken in accordance with Practice ASTM-E-883. The data will be analyzed to determine the nominal grain size, the ASTM microsize or macrosized numbers, the "Feret" diameter and grains per unit diameter. Also, data uncertainty and confidence limits will be determined.

The specimens for X-ray diffraction analysis will be taken from the average radius of the tube and will be thinned to 0.09 mm by machining and chemical milling. The analysis of the specimens will be performed in accordance to the standard method specified in ASTM-81-77 "Preparing Quantitative Pole Figures of Metals" using a molybdenum, K-alpha, X-ray source. This standard method consists of characterizing the distribution of orientations of selected lattice planes with respect to sample-fixed coordinates. The distribution will be obtained by measurement of the intensity of X-rays diffracted by the sample during both reflection and transmission. In such measurements, the detector and associated

limiting slits will be fixed at twice the Bragg angle with the incident beam, and the diffracted intensity recorded as the orientation of the sample is changed. The measured data will be corrected for background, defocusing, and adsorption and will be normalized. The data will be used to produce full pole figures for the subsequent calculation of Kearn's f-factors in the three orthogonal directions.

7.3 Specimen Description

7.3.1 Sampling Method

Samples will be taken from the ends of prototype production tubes and from both the center and ends of tubes sacrificed for destructive testing. Samples for the preparation of metallography specimens will be in the form of rings 1/2-1 inch long. They will be cut into individual specimens by longitudinally cutting the ring into segments approximately 1/2 inch circumferential dimension. Each specimen will be marked with an electric pencil to indicate the direction of drawing. Samples for the preparation of X-ray diffraction specimens will be short lengths of tubes, approximately two inches long. All cutting and machining will be performed with the cutting zone generously flooded with a continuously flowing water-based cutting/cooling fluid to prevent heating of the metal. Vise jaws used to hold the tubing and samples during cutting and machining will be padded with a soft material to prevent marring of the surfaces.

Three specimens will be selected for metallographic examination and the remainder will be identified for archival storage. After further machining and preparation described in Section 3.3, a specimen will be selected for the X-ray diffraction analysis and the remainder of the material will be retained for archival purposes. Specimens designated for each purpose will be placed in separate heavy plastic bags along with a tag containing complete identification including an identification number. This identification number will include 1) the UNC tube identification number, 2) "GS" to signify that the specimen is intended for grain size evaluation, and 3) the distance from the beginning of the

UNC tube to the location of the sample. These groups of numbers will be separated by hyphens.

7.3.2 Preparation of Metallographic Specimens

The metallographic specimens will be prepared according to ASTM-3-80. They will be mounted separately in such orientations so as to enable to the consideration of the "F" (transverse section), "G" (radial longitudinal section and "H" (tangential longitudinal section) of the tube. Each specimen mount will be marked with a vibrating pencil with the identification number of the sample described in Section 3.1 along with the suffix F, G, or H describing the orientation of the sample in the mount. Following mounting, the specimens will be rough ground, fine ground and polished according to ASTM-E-3. Note: zirconium alloys grind slowly and require sharp, fresh abrasives; silicon carbide papers, used wet, will give a fine finish. Following etching, the specimens will be thoroughly washed in water and dried. The prepared specimens will be stored in tightly covered glass or plastic containers until tested.

7.3.3 Preparation of X-Ray Diffraction Specimens

The X-ray diffraction specimen will be prepared in accordance with ASTM-E-81 using the method suggested by Tenckhof and Rittenhouse. The tube sample will be placed in a metal machining lathe and material will be removed from both the outer and inner surfaces leaving an approximately 0.3 mm thick section from the mid-radius of the tube about 20 mm long. Machining will be done in a series of light cuts to minimize deformation of the specimen. From the reduced tube section, a segment of the tube wall approximately 20 mm radial dimension and 20 mm long will be cut. The specimen segment will be chemically thinned to 0.09-0.1 mm, thoroughly cleaned with water, elastically straightened and fixed to the specimen stage of the X-ray diffractor's goniometer.

7.3.4 Sample Disposition

Following the completion of a test, test samples will be placed in plastic bags and stored in a dry location until the completion of the final report. They will then be transferred to UNC along with archival samples for storage or disposal.

7.4.0 Test Equipment

7.4.1 Equipment Description for Grain Size Evaluation

The principal equipment for the evaluation of grain size will be an optical metallographic microscope with color photographic capability. Alternatively, a metallograph capable of projecting an image on a ground glass screen may be used; however, either an integral or separate capability will be provided to produce color photographic records of the grain structure for reporting and archive purposes. In either case, the microscope and photographic device will be capable of a minimum 200x magnification. Make, model, and serial numbers of the equipment to be used will be supplied.

7.4.2 Equipment Description for X-Ray Diffraction Analysis

The diffractometer will conform to the requirements of ASTM-E-81. It will consist of 1) a X-ray source capable of producing characteristic K-alpha radiation of molybdenum, 2) slits to limit the divergence to the incident and received beam, 3) a specimen holder/goniometer assembly to position and automatically oscillate the sample in the plane of its surface without changing the orientation of the sample, 4) a scintillation X-ray detector/proportional detector, and 5) an automatic data collection system to record data for direct computer input. Alternatively data may be collected in strip charts or paper tape for manual input into the computer for data processing. The arrangement of the equipment is illustrated in Figure 1. The make, model, and serial numbers of major items of equipment used in this analysis will be supplied.

7.5.0 Test Procedure

7.5.1 Grain Size Measurements

The grain size of each specimen will be determined by the planimetric or Jeffries' procedure described in Section 9 of ASTM E-112. First, the "F" sample will be considered.

- 1) The appropriate prepared specimen will be mounted in the microscope or metallograph.
- 2) A total of 3 - 10 "representative" fields on the polished and etched surface of the sample will be chosen for examination according to the procedure described in Section 5.2 of ASTM-112.
- 3) One field selected in Step 2 will be positioned under the objective lens and a magnification will be selected that would give at least 50 grains in a circular or square 500 mm^2 inscribed on the ground glass screen of a metallograph or on a micrograph.
- 4) Focus the image sharply and, if the counting is to be performed on a micrograph, expose the film, develop it and inscribe the circle or square described in Step 3 on the finished print.
- 5) Count the number of grains totally included in the circle; count the number of grains intersected by the boundary of the surface. The sum of all grains included in the area plus 1/2 of the number of grains intersected by the boundary is defined as the equivalent whole grains, measured at the magnification used, within the area. This number is multiplied by the Jeffries multiplier for the magnification used (see Table 5 of ASTM-E-112) to give the number of grains per square millimeter.
- 6) The counting procedure described in Steps 2 and 3 will be repeated for each field selected in Step 2 and the results averaged.
- 7) The sample will be examined for intermetallic stringers or inclusions. If found, they will be characterized. The thickness of the oxide layer will be determined with a micrometric stage or comparator.

8) Steps 1 to 7 will be repeated for the "G" orientation sample.

9) Steps 1 to 7 will be repeated for the "H" orientation sample.

7.5.2 X-Ray Diffraction Analysis

Pre-Test Activities

1) Reference specimens with random orientation of the grains are required for correcting the reflection and transmission data for defocusing and absorption, respectively. If such reference specimens are not already available, they will be made using preferably the method of Kimpara, et al.* This method involves the following steps:

- . Metal powder from a zircaloy tube sample in the size range from 5 to 30 μm will be produced by filing and screening (alternatively, pure zirconium powder may be used because its X ray characteristics are identical to those of zircaloy)
- . A sample of the powder in a rubber container approximately 30 mm in diameter will be compressed to 1000 MPa in a hydrostatic press
- . The pressed sample will be sintered in a furnace at 1223 K for one hour
- . After cooling, standard specimens will be cut and mounted using the same procedure used above for the pressure tube specimens; specimens of several thicknesses approximating the tube specimens will be made.

Testing Procedures

The testing procedure described in Paragraph 5 of ASTM-E-81 will be followed.

The combined reflection/transmission method will be used with the transmission measurements being made first as recommended by ASTM-E-81. The principal steps in this procedure include the following:

- . A molybdenum K-alpha X-ray source will be selected.
- . The specimen will be mounted in the refractometer's goniometer.
- . The detector, amplifier, and pulse height selector will be set according to the manufacturers instructions.
- . The X-ray current will be adjusted to keep the counting rate in the linear region.
- . The diffracted intensity in transmission will be measured as the latitude and longitude coordinates are varied; the background on either side of peaks will be measured as a function of angle will be measured and subtracted from the peak intensities.
- . The above step will be repeated with the random standard in the specimen holder; using these results the transmission data will be corrected for absorption.
- . The tube specimen will be placed back into the specimen holder and the diffracted intensity in reflection will be measured as colatitude and longitude coordinates are varied; the background on both sides of peaks will be measured and subtracted from peak intensities.
- . The above step will be repeated with the random standard in the specimen holder; using the results, the reflection data will be corrected for defocusing.

7.5.3 Data Records

All written data will be recorded in laboratory notebooks with numbered pages. Each entry or series of related entries will be dated and initialed by the experimenter. Following submittal of the final report, the notebooks or copies of relevant pages from these notebooks along with all suitably identified stripcharts and/or DAS printouts will be transmitted to UNC for archiving or disposal.

7.5.4 Witnesses

At least one working day prior to any scheduled X-ray diffraction test described by this test procedure document, the performing organization will notify the UNC program manager of its intent to perform such a test. The performing organization will provide access to its facilities for one or more UNC representatives to witness the testing. UNC may choose to exercise or waive its right to witness any or all tests.

7.6.0 Data Analysis

7.6.1 Data Reduction Methodology

7.6.1.1 Metallographic Analysis

The data obtained in the metallographic evaluation of the three specimens taken from each sample location will be in the form of counts of grain sections per unit area. The data obtained from each specimen will be averaged and analyzed statistically to determine the limits for the 95% confidence level. These data will also be expressed in terms of nominal grain diameter, Feret's diameter, intercept size, specific surface, grains per unit volume and ASTM grain size numbers using equations provided in Annex A2 of ASTM-E-112.

7.6.1.2 X-Ray Diffraction Analysis

The data obtained on the scattered intensities during the transmission and reflection phases of the analysis will be analyzed according to procedures described in ASTM-E-81. These data will be in the form of numerical data on paper

tape, magnetic tape, or strip charts. They will be analyzed as described below by manual or computer-assisted methods or combination of the two. The transmission data, corrected for absorption and the reflection data corrected for defocusing, will be blended by scaling either all of the transmission or all of the reflection intensities so that the average of the transmission intensities along the boundary is equal to the average of the intensities along the boundary after scaling. Individual intensities along the boundary will be assigned the mean value of the corresponding reflection and transmission intensities after scaling. The intensities will be normalized to have an average value of unity. In this averaging process, each data point will be assigned a weight proportional to the solid angle which the data represents. The normalized data will be plotted on an equal area projection pole diagram. By manual or computer methods, the pole diagrams will be used to determine the Kearns f-parameters using the defining equations for these parameters.

7.6.2 Data Reported

Following data analysis, the preliminary results will be transmitted informally to UNC within 5 working days. For the metallographic analysis, these results will include nominal grain sizes in the radial, longitudinal and transverse directions and the characterization of any inclusions found in the grain structure. For the X-ray analysis, the texture of the samples as described by the Kearns f-parameters will be reported.

At the conclusion of this testing, a final report describing these tests will be issued. This report will include the following information:

- . Description of the apparatus used in each type of test
- . Typical color photomicrographs
- . Color photomicrographs of unusual grain structures
- . Summaries of grain size data
- . Tabulations of results of grain size results in terms of nominal diameters, Feret's diameter, intercept size, specific surface, grains per unit volume and ASTM grain size number.
- . Statistical data on grain size information
- . Pole diagrams for each sample
- . Kearns f-parameters (f_r , f_t , and f_l) for

- each sample.
- . Sample calculations.

REFERENCES:

Teckhoff, E. and P. L. Rittenhouse, APPLICATIONS-RELATED PHENOMENON IN ZIRCONIUM AND ITS ALLOYS, ASTM STP 458, American Society for Testing and Materials, 1969.

Kimpara, M. K. Fujita, T. Kakuma, and N. Nagai, ON THE TEXTURE MEASUREMENTS FOR ZIRCALOY CLADDING TUBE, Zirconium in the Nuclear Industry: Sixth International Symposium, ASTM STP 824, American Society for Testing and Materials, 1984.

APPENDIX

REFERENCED ASTM STANDARDS