**Nuclear Energy Research Initiative (NERI) Program
Continuous Fiber Wound Ceramic Composite (CFCC) for Commercial
Water Reactor Fuel
DE-FG03-99SF21887
Gamma Engineering
Technical Progress Report
April 1, 2000**

Narrative:

Our program began on August 1, 1999. As of April 1, 2000, the progress has been in materials selection and test planning. Three subcontracts are in place (McDermott Technologies Inc. for continuous fiber reinforced ceramic tubing fabrication, Swales Aerospace for LOCA testing of tubes, and Massachusetts Institute of Technology for In Reactor testing of tubes).

With regard to materials selection we visited McDermott Technologies Inc. a number of times, including on February 23, 2000 to discuss the Draft Material Selection and Fabrication Report. The changes discussed at this meeting were implemented and the final version of this report is attached (attachment 1). McDermott Technologies Inc. will produce one type of tubing: Alumina oxide (Nextel 610) fiber, a carbon coating (left in place), and alumina-yttria matrix. A potentially desirable CFCC material of silicon carbide fiber with spinel matrix was discussed. That material selection was not adopted primarily due to material availability and cost. Gamma Engineering is exploring the available tube coatings at Northwestern University as a mechanism for reducing the permeability of the tubes, and thus, will use coating as a differentiating factor in the testing of tubing in the LOCA test as well as the In-Reactor Test. The conclusion of the Material Selection and Fabrication Report lists the possible coatings under evaluation.

With regard to Test Planning, the MIT and Swales Aerospace have submitted draft Test Plans (attachment 2 and 3). MIT is attempting to accommodate an increased number of test specimens by evaluating alternative test configurations. Swales Aerospace held a design review at their facilities on February 24, 2000 and various engineering alternatives and safety issues were addressed. The final Test Plans are not expected until just before testing begins to allow for incorporation of changes during "dry runs."

Issues and concerns:

We are somewhat behind schedule, but expect that we will have our materials selection and fabrication work done within the framework of phase one, without difficulty. Specimen testing is not scheduled until phase 2, which allows plenty of lead-time for fabrication.

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Gamma will be formulating an additional contractual relationship with Northwestern University of tubing coating services. We do not expect the tubing coating to increase the cost of the overall project or delay the overall project schedule.

Cost Performance:

See attached chart. Cumulative spent through March is \$ 92,361, lower than our projected \$192,862 due to delays in subcontracting, but we expect expenses to increase over the next few months as subcontractors complete their tasks.

Status Summary:

Phase 1 – 12 months

Milestone/Task Description	Planned Completion Date	Actual Completion Date
Materials Selection Report	November 15, 1999	March 14, 2000
Shipping Papers	May 1, 2000	
Fabrication Report	June 1, 2000	
Topical Report (the above 3 items)	August 5, 2000	

Phase 2 – 6 months

Milestone/Task Description	Planned Completion Date	Actual Completion Date
Loss of Coolant Accident Test Report	November 1, 2000	
In-Pile Testing of CFCC Cladding Report	December 1, 2000	
Final Technical Report (Describing the Feasibility of using CFCCs as fuel Cladding)	April 1, 2001	

Actual Versus Projected Cost for "CFCC Cladding of Commercial Water Reactor Fuel"

PiNERI Project CFCC Fuel Cladding
Date: 8/18/99 8/13/99

Task

Summary

Rolled Up Progress

Progress

Rolled Up Task

Milestone

Rolled Up Milestone

McDermott Technology, Inc.

a McDermott company

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GAMMA ENGINEERING CORP

Contract Research Division

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March 14, 2000

Continuous Fiber Ceramic
Composite Cladding for Commercial
Water Reactor Fuel
MTI: CRD 1405

Ms. Bonnie Packer
Gamma Engineering Corp.
15815 Crabbs Branch Way
Rockville, MD 20855

Dear Ms. Packer:

Subject: Material Selection and Fabrication Report

Attached is McDermott Technology's **Material Selection and Fabrication Report** for the referenced contract. This report has been modified to include Gamma Engineering's comments from the February 23, 2000, meeting at McDermott's Lynchburg, Va., facilities. If there are any questions about this submittal, please contact Dr. Wagner at (804) 522-5697.

Sincerely,

McDERMOTT TECHNOLOGY INC.
a McDermott company

J.P. Doran
Senior Contract Manager

Attachment

c: H.H. Davis, MTI, w/o attachment
E.A. Barringer, MTI, w/o attachment
R.W. Goettler, MTI, w/o attachment
R.A. Wagner, MTI, w/attachment

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**Continuous Fiber Ceramic Composite
Cladding for Commercial
Water Reactor Fuel**

Material Selection and Fabrication Report

PREPARED BY

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SUBMITTED BY

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MARCH 14, 2000

PREPARED FOR

**GAMMA ENGINEERING CORPORATION
ROCKVILLE, MARYLAND
CONTRACT NO. 99-41-001
MTI CONTRACT NO. CRD 1405**

Material Selection and Fabrication Report

The focus of this report is to evaluate candidate ceramic materials for potential use as nuclear fuel cladding. The relevant experience base of McDermott Technology Inc. is primarily in the area of oxide/oxide composites produced by filament winding and densifying by sol-gel methods. Since resource limitations do not permit material development activities, MTI is recommending materials (reinforcement fibers, matrix powder, matrix precursors) from the experience base of our DOE Continuous Fiber Ceramic Composite (CFCC) program and other company programs.

Fabrication Process

The fabrication process will be described first because it further constrains the material selection process. Continuous fiber ceramic composites (CFCCs) will be fabricated by filament winding a preform followed by a two step densification process. Prior to filament winding, the mandrel will be prepared by wrapping with a suitable filter paper. An En-tec computer controlled filament winder will be used to produce a helical winding pattern on a stainless steel mandrel (0.38 inches O.D. by 26 inches long working length). Sufficient closures will be applied to achieve a diameter of 0.50 +/- 0.05 inches.

In the first densification step, the voids between the fibers will be filled by pressure casting a slurry of fine powder through the cross-section of the part. In order to fill the voids in the preform, the particle size of the slurry must be much smaller than the fiber diameter. Typically, powders with a mean size of 0.2 to 0.5 μ are used to fill the voids between the Nextel fibers. The powder slurry is prepared by ultrasonic dispersion in water to break up agglomerates. Figure 1 illustrates the elements of the pressure casting process and shows a partially filled preform. In this process a filter cake develops at the filter paper surface and builds up through the preform. The water passes through the filter paper into the hollow mandrel and drains from the system. Pressurizing the slurry increases the casting rate. Careful control of the slurry particle size distribution can increase the initial matrix density and save down-stream processing steps. Following the pressure casting process, the preform is dried on the mandrel and pre-fired to burn off the filter paper to facilitate mandrel removal.

Final densification of the component is achieved by vacuum impregnation with a liquid polymeric precursor to form additional matrix material within the voids of the pressure cast powder matrix. Approximately 10 to 15 polymer impregnation/drying/firing cycles are required to achieve an 80% dense composite.

Composition Considerations

The material selection is guided by considering the corrosion resistance, thermal conductivity, nuclear cross section, radiation damage; and finally material availability (i.e. fiber, matrix powder, matrix precursor, and chemical vapor infiltration precursors).

Corrosion Resistance

Figures 2-4 include the phase diagrams for silica/water, alumina/water, and chrome oxide/water, respectively. At the fuel cladding environmental conditions (343 °C (650 °F) and 2000 psi) each of these phase diagrams indicate that the oxy-hydroxide is the stable phase for many of the candidate metal oxides. At temperatures above about 400 °C, the oxide is the stable phase. Autoclave exposure test results for several oxides were reported previously by Ramey¹. These results are summarized in Table 1. The autoclave test conditions were 72 hours at 554°F with a saturation pressure of about 1000 psi. The data for the alumina-chrome series were obtained after 300 hours at 650°F and 2200 psi. The alumina-chrome data are given as percent weight loss per day because the sample size was not reported.

Table 1 Corrosion results for candidate cladding materials.

material	Additives	impurities	Phase content	Grain size	Weight loss w/o O ₂ (mg/dm ² /day)	Weight loss w/O ₂ (mg/dm ² /day)
Al ₂ O ₃ 96%		SiO ₂ , CaO	α- Al ₂ O ₃	1-10	80	80
Al ₂ O ₃ 99%			α- Al ₂ O ₃	1-20	13	16
Al ₂ O ₃ 99.5%			α- Al ₂ O ₃	1-20	11	10
Al ₂ O ₃ 99.99%			α- Al ₂ O ₃	2-10	1	0.4
Al ₂ TiO ₅						
ZrO ₂	3 m/o Y ₂ O ₃		tetragonal	0.5	10	10
ZrO ₂	6 m/o Y ₂ O ₃				0.1	0.2
ZrO ₂	8 m/o Y ₂ O ₃				0.03	0.1
Al ₂ O ₃					0.08*	-
Al ₂ O ₃	5 m/o Cr ₂ O ₃				0.02*	-
Al ₂ O ₃	10 m/o Cr ₂ O ₃				0.006*	-

Notes: * weight loss expressed as percent/day

Because impurities such as silica and lime are relatively soluble under the conditions of interest and tend to segregate along grain boundaries, hydrothermal corrosion often leads to disintegration of the component.

Continuous Fibers

Table 2 lists the candidate continuous fibers that have been utilized at MTI in composite fabrication. As indicated in the Table 1, the 3M Nextel 610 fiber is the preferred reinforcement fiber for the proposed fuel cladding. MTI is working with 3M to develop alumina-zirconia and yttria-alumina-garnet (YAG) but these are not presently available for use in fabricating components. The Nextel 720 and Altex both contain approximately 15 wt/percent silica and are therefore likely to exhibit significant degradation in conditions found in reactors and steam boilers.

Table 2. Candidate reinforcement fibers.

Fiber	Supplier	Composition	Comments
Nextel 610	3M	Alumina	Best option
Nextel 720	3M	Alumina-mullite	Silica portion soluble in saturated steam conditions
Altex	Sumitomo	Alumina-silica	Silica portion soluble in saturated steam conditions

Fiber Coating

The interface between the reinforcement fiber and the matrix must be controlled in order to ensure composite-like mechanical behavior. If the reinforcement fiber reacts or otherwise bonds to the matrix, then the composite will behave like a monolithic material and fail catastrophically. MTI has implemented a fiber coating process that utilizes immiscible liquids to minimize coating bridging between the individual fibers in a fiber tow bundle. A fugitive carbon coating based on furfuryl alcohol is recommended for this program. MTI has coated over 40 pounds of Nextel 610 fiber using this system. Other coatings are under development and could be utilized in future work.

Matrix

As described above, the matrix is derived from the combination of pressure cast powders and polymer or sol-based precursors. Matrix materials under consideration include: alumina, yttria-alumina, zirconia, and magnesium-aluminum spinel. Each of these is available as fine powders suitable for pressure casting. Liquid or sol-based precursors are available for the alumina, yttria-alumina, and zirconia systems. MTI has only limited experience with spinel precursors. MTI has the most composite fabrication experience with alumina and yttria-alumina matrix systems. Although the thermal conductivity of fully dense alumina or yttria-alumina systems are higher by about an order of magnitude compared to zirconia, the 15 to 25% porosity of the MTI composites may reduce

the difference in the finished composite. Thermal conductivity results for an early version of the MTI alumina composite were in the range of 1 to 2 W/mK. Because thermal conductivity is such a critical property, we will restrict the matrix selection to the yttria-alumina system.

Exterior Coating

Both the corrosion resistance and permeability of these compositions will be compromised by the residual porosity of the composite. An external coating should be considered as a means to seal the composite in order to decrease the permeability of both water and fission gases. Northwestern University has successfully deposited dense alumina coatings by a chemical vapor deposition (CVD) process. MTI recommends that at least some of the corrosion samples be coated to evaluate the effectiveness of this approach.

Radiation Damage and Absorption Cross Section

Neutron irradiation damage in dense oxide materials may cause point defect agglomeration to form voids leading to swelling of the component and significantly decreased thermal conductivity. Kinoshita et al. compared the radiation damage in alumina and magnesium-alumina spinel (MgAl_2O_4). The expected fluence in a fission reactor is approximately an order of magnitude below the critical fluence for spinel and equal to or above the critical fluence for void formation in alumina. Depending on the irradiation temperature, the thermal conductivity of alumina decreased 50 to 75% according to Thorne and Howard. The impact of irradiation damage induced swelling in composites containing 15 to 20% porosity is not known. No hydrothermal corrosion data for spinel were found.

The nuclear absorption cross section of the candidate materials must also be considered in the selection process. Table 3 lists the some of the materials of interest. Although chrome oxide appeared to improve the hydrothermal corrosion resistance of alumina, the relatively high cross section of Cr limits its use. Furthermore, the high cross section of carbon will require a complete burnout of the carbon coating on the continuous fibers. Finally, the high cross section of hafnium must be considered since it is a common impurity in zirconia.

Table 3. Macroscopic absorption cross sections of elements contained in candidate materials.

Element	Macroscopic Section cm^{-1}	Cross
Zr	0.008	
Al	0.015	
Mg	0.003	
Cr	0.255	
Y	0.049	
Si	0.008	
Hf	4.710	
C	32.000	

Recommendations

Based on the discussion above, eight tubes (2 ft long) will be fabricated using the following composite system:

Nextel 610 fiber, carbon coating, yttria-alumina matrix.

The base composite system will be coated with one or more of the following CVD coating candidates:

- 1) 10 wt% Cr_2O_3 -alumina (thickness of 2 - 4 μ).
- 2) Pure Al_2O_3 (thickness 2 - 4 μ) plus flash coating of ZrO_2 .
- 3) Pure Al_2O_3 (thickness 2 - 4 μ)

References

1. R. R. Ramey, Literature Review Ceramics For Electrode Insulators, Report Number RDD:90:7289:01, 1990
2. Kinoshita, C., et al. "Why is magnesia spinel a radiation-resistant material?", Journal of Nuclear Materials, 219, 143-151, 1995.
1. Thorne, R. P., and Howard, V. C., Changes Induced in Polycrystalline Alumina by Fast Neutron Irradiation", Proc. Brit. Ceram. Soc. 7, 439-447, 1967.

Figure 1. Schematic diagram of pressure cast matrix densification process.

Figure 2. Silica-water phase diagram.

Figure 3. Alumina-water phase diagram.

Figure 4. Chrome oxide-water phase diagram.

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CONTRACT NO.: 99-41-002

TASK NO.: 6536

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Gamma-LOCA Test Plan

SAI-PLAN-375

12/30/99

Draft

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SIGNATURE PAGE

This is a controlled document. Any changes require the approval of the Configuration Control Board.

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Gamma Engineering: _____
Date

DOCUMENT CHANGE RECORD

REVISION	DESCRIPTION	DATE	APPROVAL
-	Draft	12/30/99	jlbaker

TABLE OF CONTENTS

1.0	SCOPE	4
2.0	APPLICABLE DOCUMENTS	4
3.0	REQUIREMENTS.....	4
3.1	Apparatus	5
3.1.1	Design	5
3.1.2	Fabrication	7
3.1.3	Calibration.....	7
3.2	Health and Safety	8
3.2.1	Plans and Procedures	8
3.2.2	Zircaloy Heat and Quench	8
3.2.3	Zircaloy Machining.....	8
3.3	Testing.....	9
3.3.1	Specimen Characterization	9
3.3.2	Ceramic Testing	9
3.3.3	Zircaloy Testing	10
3.4	Reporting.....	10
3.4.1	LOCA Test results	10
3.4.2	DOE Report	10

Attachments

1	Program Schedule
2	Electrical Schematic
3	LOCA Apparatus, Plan View
4	LOCA Apparatus, Top View
5	LOCA Drop Mechanism
6	Kanthal MS26 Drawing
7	Digital Photo

1.0 SCOPE

Swales Aerospace is under subcontract to Gamma Engineering's DOE-NERI program DE-FG03-99SF21887-A000, entitled "Continuous Fiber Ceramic Composite (CFCC) cladding for Commercial Water Reactor Fuel". Swales will assemble and operate a Loss of Coolant Accident (LOCA) apparatus to simulate an Emergency Core Cooling System (ECCS) quench. The specific objective of this effort will be to heat and quench ceramic specimens as candidate replacements for Zircaloy cladding of nuclear fuel rods. Pre and post quench test characterization of the specimens will be performed. Temperature profiles of the specimens and the water quench will be reported.

2.0 APPLICABLE DOCUMENTS

DOCUMENT NUMBER	TITLE
99-41-002	Gamma Engineering Purchase Order (P.O.)
SAI-HAS-001	Swales Health and Safety Plan
SAI-HAS-011	Swales Electrical Work Safety Rules
SAI-HAS-023	Swales Handling and Distribution of Nitrogen Gas
SAI-HAS-036	Swales Oven Operating Procedure
SAI-HAS-043	Swales Emergency Evacuation Plan

3.0 REQUIREMENTS

This Test Plan will describe and illustrate the design, fabrication, and calibration to be used for the Swales LOCA apparatus. The plan shall define the CFCC ceramic and Zircaloy specimens to be tested, and the temperatures which they will be exposed to prior to quench. The specimen pre-test and post-test characterization will be defined. The relevant health and safety considerations will be discussed. The reporting of test results and data will be defined. The overall schedule for this project is included as Attachment 1.

3.1 Apparatus

3.1.1 Design

3.1.1.1 Energy Balance

Energy balance calculation has been made to determine rise in temperature of the water upon quench. The equilibrium temperature of 1.5 liter of water should not rise by more than 5 degree F in case of 15 gram ceramic specimen, or by more than 2 degree F in case of 16 gram Zircaloy-4 specimen. Rate of heat transfer calculation shows that the specimen will cool to below 250 F in approximately 15 minutes.

3.1.1.2 Apparatus Design

The overall configuration of the LOCA apparatus is shown as "Electrical Schematic" as Attachment 2. At the heart of the chamber is a Kanthal MS26 mini furnace. This will be driven by 1200 Watt DC power supply controlled using desktop PC with Labview software. The Kanthal furnace can be run up to 2700-2800 F with full power applied. This will be adequate to get 15 gram specimen up to 2500 F. The soak time to get to full temperature has not been calculated but is expected to be on the order of 1-2 hrs.

The Kanthal MS26 furnace will be mounted within a 304 stainless steel (SS) tube, 12-inch diameter x 0.180 thick. The furnace will be mechanical fastened to cross plates that will be welded to interior of tube. This will enable change-out of MS26 if necessary. The cross plates will be 0.187 inch thick 304 SS. The exterior of the tube will insulated with 1/16 inch thick fiberglass cloth. The vessel will have top and bottom 304 SS covers, 0.187 inch thick. The bottom cover (base) will be removable via cam lock or toggle clamps. The top cover will be clamped to a flange that is welded to the cylinder. This arrangement is shown in "Plan View" Attachment 3, and "Top View" Attachment 4. The detail of the Kanthal MS26 can be seen in Attachment 6.

A simple rail system with alumina sliding door will be assembled within the 304 cylinder. Two stainless steel extension springs will hold the door in the shut position and a 1/16" SS cable will be used to manually pull the door open. The springs will quickly pull the door closed. This is illustrated in Attachment 4 and "Drop Mechanism" Attachment 5. This mechanism will allow the test specimens to drop into a stainless steel (S.S.) beaker of 1.5 liter (1500 grams) water. Distilled water will be heated and maintained at 200 F. The water will be heated using a small resistance heater plate, driven by an SCR controller. At this time, simple fittings to provide Nitrogen purge are planned, but the chamber could be made pressure tight if necessary. Argon may be used instead of nitrogen during Zircaloy testing.

The ceramic test specimens have been identified as 3.0 inch long x 0.5 inch diameter, and .060 inch wall thickness. The Zircaloy specimens will be same except 0.026 wall thickness. The specimens are therefore approximately 15 grams. The water will stay below boiling, however some steam will be generated by local vaporization as the specimen settles in the water.

Swales will use commercially available Alumina (Al_2O_3) tubes for performing verification and calibration of the chamber. Verification refers to the general capability of the chamber to come up to temperature (approximately 2500 F) at a reasonable ramp rate and release of the tubular specimens into the beaker of water. Calibration refers to 1st establishing a correlation between fixed furnace thermocouples and test specimen thermocouples. Secondly a correlation will be made between specimen thermocouples and IR sensor. This is discussed in more detail in section 3.1.3.

3.1.1.3 Equipment List:

- Kanthal MS26 Furnace(primary and backup)
- Xantrex XFR 40-30 DC power supply (RS232 interface)
- PC with Labview software
- 304 Stainless Steel:
 - 12 inch diameter x 20 long vessel
 - 3/16 inch inch plate for flanges
 - 1" x 1" & 2"x 2" angle for drop mechanism
- Omega Type R (Pt/Rh) thermocouples and high temp cement
- Fluke Netdac Data acquisition unit
- Raytek FA2B-CF11 Fiber optic Infrared temperature sensor
- Coors Ceramics AD-998 alumina tube for calibration
- Polarware 3W stainless beakers (qty 2)
- 250 Watt Cartridge heater plate for water
- Omega SCR (110v AC) for heater plate control
- Type T thermocouples for water and air(nitrogen purge) temp
- Supply tank, regulator, line and fittings for compressed dry nitrogen (LN2)
- Zircar RS-99R alumina plate for drop mechanism
- 304 SS Springs and cable for drop mechanism
- Cam locks and spring clamps for vessel flange release

3.1.2 Fabrication

The Swales 5021 machine shop will fabricate the 304 stainless steel chamber, flanges, and drop mechanism components. The Kanthal furnace and drop mechanism will be assembled in 5021 assembly area.

All electrical connection, temperature instrumentation, and calibration will be performed at the Swales Thermal Systems (STS) building at 11313 Frederick Ave., Beltsville, MD.

3.1.3 Calibration

3.1.3.1 Specimen to Furnace Correlation

The apparatus will be operated using Alumina tubing as a "dummy" material. A sample of Coors AD-998 tubing was obtained for this purpose. Type R thermocouples (Pt/Rh) will be cemented to the top and bottom of alumina specimen. Thermocouples will be permanently cemented to the interior element of Kanthal furnace at top and bottom, and to the middle of external shroud. A correlation of furnace temperature to specimen temperature will be developed. Plotting the heatup profiles using PC Labview will be used for this.

3.1.3.2 Drop Mechanism Verification

After a specimen to furnace temperature correlation is achieved, but before the IR sensor is installed, the drop mechanism will be proven out at elevated temperature. A specimen heat-up to 1000 F and tube drop will be performed with alumina sample. This will be done with the nitrogen purge on and will also verify that steam generated by specimen drop does not get into Kanthal furnace and cause failure from shock. This trial run verification will be repeated at 1500 F, 2000 F, and 2500 F. This verification phase will also determine time required to bring the water bath up to 200 F steady state.

3.1.3.3 IR Sensor Calibration

Specimen thermocouple data will also be compared to IR sensor data to be sure IR sensor is reading the specimen properly. The surface emissivity of the ivory colored alumina sample is expected to be very representative of the experimental CFCC ceramic test specimens. The Raytek FA2B has surface emissivity adjustment capability from 0.1 to 1.0 in 0.01 increments. Emissivity adjustment and rerun will be done as necessary to bring the thermocouple and IR readings within 25 degree F.

- Accuracy of Type R (Pt/Rh) thermocouples is $\pm 0.25\%$. At 1500 F, thermocouple measurement is ± 4 degree F. At 2500 F, measurement will be within 6 degree F.
- Accuracy of the Raytek FA2B optic thermometer is $\pm 0.3\%$. At 2500 F, measurement is within 7.5 degree F of actual temperature.

A separate TC instrumented Zircaloy calibration run up to limited temperature (eg: 1500 F) will need to be performed. Emmisivity adjustment will be made to attempt IR sensor measurement of the Zircaloy up to maximum exposure temperature. If agreement between thermocouple and IR sensor cannot be achieved, the furnace to Zircaloy specimen temperature correlation will be relied upon as certain.

3.2 Health and Safety

3.2.1 Plans and Procedures

The Program Manager (Mr. Jeff Baker) has reviewed all the referenced Swales Health and Safety documents. Mr. Baker will be the Program Health and Safety Officer and implement any and all necessary measures to be in compliance with these plans and procedures. A copy of these documents will be kept on hand at the LOCA test site. This test plan with design sketches has been reviewed with the Swales Company Health and Safety Officer (Mr. Ron Bettini). Any incident or mishap will be reported to the Swales Company Health and Safety Officer.

3.2.2 Zircaloy Heat and Quench

There are no inherent risks associated specifically with testing of the alumina or CFCC ceramic specimens. These ceramics are very inert and stable to over 3000 F. The Zirconium based Zircaloy-4 alloy however does require some special precautions. The MSDS for Zircaloy has been reviewed. Zircaloy can undergo a heat producing reaction with oxygen at high temperatures. This necessitates inert gas blanketing (eg: nitrogen purge) to preclude specimen fire from reaction with oxygen. The nitrogen purge system on the LOCA apparatus will be vented to an existing Swales fume hood or to a project specific roof vent. There will be no other flammable materials used in apparatus construction. A Type D portable fire extinguisher (dry chemical for flammable metals) and a 50 lb bag of dry sand will be kept at the LOCA test site in case the nitrogen purge is not adequate. The Zircaloy will also generate a slight amount of hydrogen along with some steam vapor upon quench. This will subside quickly and be carried away with the nitrogen purge. It is important to keep in perspective that the Zircaloy specimen is only 15 grams being dropped into 1500 gr (1.5 liter) of distilled water. The energy balance calculation shows only a 2 degree F rise in water temperature.

3.2.3 Zircaloy Machining

It is also important to not create small chips or powder of Zircaloy that can react with oxygen. The Zircaloy specimens will be supplied to the 3.1-inch maximum length as stated in the specimen "specification" described in "testing" section 3.3. There will be no machining of Zircaloy at Swales.

3.3 Testing

All testing will be performed at the Swales Thermal Systems (STS) building at 11313 Frederick Ave., Beltsville, MD. Jeff Baker, Stuart Whitelock, and/or other trained technicians as necessary will carry out testing. The Swales LOCA apparatus can accept specimens up to 3.100 inch in length and up to 0.75 inch in diameter. Interior dimensions of the KANTHAL MS26 furnace drive this size limitation. Swales will concur by signature with these dimensions in the "specification" document to be reviewed for Gamma Engineering. The temperature ramp and hold of the water bath will be controlled by a programmable Omega SCR controller. As the water bath is raised to 200 F, the Kanthal furnace will concurrently ramp the specimen to the desired test temperature. A desktop PC using Labview software controls the Kanthal furnace, driven by the DC power supply.

3.3.1 Specimen Characterization

3.3.1.1 Pre Test Characterization

The following measurements will be taken prior to heat and quench

- Weight to within X.XX grams
- Dimensions (ID, OD, length) to within 0.001 inch
- Digital Photos (alumina sample shown as attachment 7)
- Stereomicroscope inspection at 7-30X
- The pre-test weight of water in beaker will be recorded within X.XX grams.

3.3.1.2 Post Test Characterization

- Weight to within X.XX grams
- Dimensions (ID, OD, length) to within 0.001 inch
- Digital Photos
- Stereomicroscope inspection at 7-30X

If any significant anomalies are visually apparent, Swales can send the specimens out for photomicrographs. The post-test weight of water in beaker will be recorded.

3.3.2 Ceramic Testing

Experimental CFCC ceramic specimens are due from McDermott approximately June 1, 2000. McDermott is producing two candidate CFCC tubes. The Pre and Post quench characterization will be performed for the following quenches.

- **Ceramic A**

- 2 specimens at 1000 F
- 2 specimens at 1800 F

-2 specimens at 2500 F

- **Ceramic B**

-2 specimens at 1000 F

-2 specimens at 1800 F

-2 specimens at 2500 F

3.3.3 Zircaloy Testing

After the Ceramic specimens are fully tested, Zircaloy-4 specimens provided by Gamma Engineering will be quench tested. The Pre and Post quench characterization on the following specimens is currently planned:

-1 specimen at 1000 F

-1 specimen at 1500 F

-1 specimen at 2000 F

3.4 Reporting

3.4.1 LOCA Test results

Within 30 days of completion of testing, a report will be submitted to Gamma Engineering. The temperature profiles from all runs will be provided. Temperature profiles will include furnace, specimen, water, and nitrogen purge. Electronic copies will be available as required. The report will include the **pre and post** test characterization of all specimens.

-Weight to within X.XX grams

-Dimensions (ID, OD, length) to within 0.001 inch

-Digital Photos

-Stereomicroscope inspection at 7-30 X

If any significant anomalies are visually apparent, Swales can send the specimens out for photomicrographs. The pre and post test weight of water will also be reported for each run.

3.4.2 DOE Report

Swales will review Gamma Engineering's final report for DOE in the December, 2000 time frame.

GAMMA - LOCA Test

Date Printed: Fri 1/21/00 6:35 PM

Gamma - LOCA Apparatus

Electrical Schematic (A2)

Rev - 12/13/97

Gamma - LOCA Apparatus Plan View (A3)

Rev - 12/15/9

Gamma - LOCA

Top View
(A4)

Rev - 1/25/

1:2 Scale

Gamma - LOCA

Drop Mechanism
(A5)

Rev-1/25/00

Spring
2 plc

Zircar
RS-99 R
Aluminum

0.50"
thick

Omega-
Bond
Cement

1x1
304
angle

Mycalex 400
Washers (1/8" thick), 4 plc

1/4-20 x 1/2" lg cap screws

Crossbars, 3/16" 304

1/8" spacers

1/4-20 x 1" cap screws

2 x 2 x 1/8" 304 angle

$$2500^{\circ}\text{F} = 1370^{\circ}\text{C}$$

16 A × 19 V @ 1500°C
27 Amp max load
200°C outside

Date: 930518

Scale: 1:1

Drawn by: LGJ

MS 26

Kantha /
Mini
Superthal

Attachment 2

Digital Photo of Alumina Sample

DRAFT

RECEIVED

APR 03 2000

Test Plan for In-reactor Corrosion Test of Oxide-based CFCC Tubes at the MIT Nuclear Reactor Laboratory

Prepared by Dr. G. Kohse
MIT Nuclear Reactor Laboratory
Nuclear Engineering Dept.

March, 2000

1. *Introduction*

This test plan describes work to be performed at the MIT Nuclear Reactor Laboratory for Gamma Engineering Corporation as part of Gamma Engineering's Department of Energy Financial Assistance Award, DE-FG03-99SF21887-A000. The major portion of the work at MIT falls under Task 3 of the award: in-pile testing. The plan is divided into four sections. This introduction is followed by a description of the test facility design, including the in-pile autoclave, the specimen dimensions that can be accommodated and the out-of-core support systems. The third section describes the test program, including the pre- and post-irradiation tests that will be performed, and the protocol for the irradiation test. The fourth section specifies instrument calibration procedures and a laboratory Health and Safety Plan.

2. *Test Facility Design*

The test facility to be used in this program is based on test facilities that have been operated previously in the MITR-II, a 5 MW research reactor operated by the MIT Nuclear Reactor Laboratory. The facility consists of: an autoclave that will be installed in an in-core position in the reactor; an aluminum "thimble" to isolate the autoclave from the reactor coolant environment (light water at $\sim 50^{\circ}\text{C}$ and atmospheric pressure); an external primary loop providing coolant pumping and heating for the in-core autoclave; a bypass system to control coolant chemistry and pressure; and control and alarm systems. Each of these systems is described in this section of the test plan.

A cross-section of the MITR-II illustrating the installation of the in-core components is given in Figure 1. Note that the autoclave, in its isolating thimble, is installed through the reactor top lid into fuel element position in-core where the fuel element has been replaced by a dummy element with a central hole sized to accommodate the thimble and allow for coolant flow. All connections to the autoclave and thimble are made above the reactor lid.

The in-core autoclave is constructed from Grade 9 titanium alloy tubing 1.5 inches outside diameter with .080 inch wall thickness. This tubing is able to withstand the required operating temperature and pressure under ASME Code design rules. Within this autoclave, cladding specimens with 0.5 inch outside diameter, 1/16 inch wall thickness by 3 inches long are positioned. The specimens are mounted in pairs as shown in Figure 2. Each specimen pair is held in a titanium spacer and is surrounded by a cylindrical titanium shroud that creates an annulus for coolant downflow around the specimen region. Five sets of specimens can be accommodated over a length of about 16 inches in the central region of the core. The total core height is about 22 inches, but greater flux uniformity can be obtained if the ends of the core are not used for specimen irradiation. Additional specimens can be positioned in the above core region for comparison, if desired. A total of 10 in-core specimen positions is provided using this design, the test matrix to be agreed by Gamma and MIT will specify what CFCC materials and comparison Zircaloy-4 specimens will be installed at each position. The specimen stack

will be pre-assembled and installed in the autoclave before installation of the autoclave in the MITR-II.

The out-of-core systems consist of a high temperature recirculating loop providing coolant to the in-core autoclave at the desired temperature, and a low-temperature bypass system that controls the coolant chemistry and pressure. The primary flow loop is illustrated schematically in Figure 3. The recirculating pump is a magnetic drive, sealless pump manufactured by Autoclave Engineers. It will provide a flow rate of approximately 5 gallons per minute to the in-core autoclave. The heater is two tubes passing through an electrically heated lead bath. This design provides desirable thermal inertia, which eases the thermal and pressure control of the loop. Heated water is pumped into the autoclave where it flows down over the specimen stack shroud as shown. The coolant then flows upward over the in-core and out-of-core specimens before returning to the external loop. Approximately 0.2 gallons per minute of coolant are added to and extracted from the primary loop by the chemistry and pressure control system described below.

Figure 4 is a detailed schematic of the chemistry and pressure control system. Instrumentation is provided to measure dissolved hydrogen and oxygen (H_2 , O_2) and conductivity (μ) of the coolant. Full flow ion exchange (IX) of the loop letdown and bypass ion exchange of the coolant reservoir is used to maintain coolant purity. The coolant reservoir is also continuously sparged with an appropriate gas or gas mixture to maintain the desired dissolved gas concentrations. Pressure control is achieved using the positive displacement charging pump (manufactured by American Lewa) and a backpressure regulator that controls the letdown flow from the primary loop. Two heat exchangers are used to cool the loop letdown. The first is a regenerative heat exchanger that also preheats the charging flow, while the second is cooled by MITR-II secondary coolant and ensures that the exiting coolant is adequately cooled to avoid damage to the ion exchange resins and coolant instrumentation.

The systems described above are designed to provide a coolant environment that closely simulates that of a typical pressurized water power reactor (PWR). Table 1 lists the planned values of the important parameters for the in-core test.

Table 1. Planned values for in-core test parameters

Parameter	Value
Fast neutron flux ($E > 1 \text{ MeV}$)	$5 \times 10^{13} \text{ n/cm}^2\text{-s (avg.)}$
Thermal neutron flux	$2 \times 10^{13} \text{ n/cm}^2\text{-s (avg.)}$
Gamma dose rate	10^9 R/h
Temperature	300 C
Boric acid concentration	800 ppm B
Lithium hydroxide concentration	1.4 ppm Li
Hydrogen concentration	20 cc/kg

Note that the planned boric acid and lithium hydroxide concentrations will result in a pH (300 C) of 7.0. This is at the low end of the pH range generally used in currently

operating PWRs. The boron concentration of 800 ppm is representative of mid-cycle boron concentrations. Note that other boric acid and lithium hydroxide concentrations can be used if mutually agreed between Gamma and MIT.

3. *Irradiation Test Program*

As discussed above, the matrix of specimens to be tested will be agreed between Gamma and MIT subject to the limitations on total in-core specimen numbers imposed by the facility design. MIT will perform the tests listed in Table 2 on each specimen before and after irradiation. If Gamma provides an archive specimen of each material type (not to be irradiated), these specimen will also be tested.

Table 2. Pre- and post-irradiation tests for clad specimens.

Test	Parameters measured	Comments
Visual inspection	Color/decoloring Physical damage	All specimens, photographic documentation where required.
Weight and dimensions	Pre- and post-irradiation weight, length, diameter and wall thickness	All specimens, post-irradiation dimensions may be measured optically (dependent on residual activity levels).
SEM	Surface condition (particle size, porosity, cracking, blistering) Section microstructure (voids, cracks)	All specimen outer surfaces observed, section microstructure examined for selected high-interest specimens as time permits
Gamma spectroscopy	Specimen activity by species	Selected specimens for fluence confirmation, contamination characteristics

The irradiation test itself is planned for days of reactor operation. Due to the operating schedule of the reactor, these test days will not necessarily be continuous. During short reactor outages, thermal and chemistry parameters will be maintained as given in Table 1. For longer reactor outages, standby conditions will be agreed between Gamma and MIT. Table 3 summarizes the parameters to be measured during the irradiation test, and specifies the frequency of measurement.

Table 3. Parameters to be measured during the irradiation test

Parameter	Measurement Type	Minimum Frequency
Specimen temperature	On-line, computer logged	10/h
Loop pressure	On-line, computer logged	10/h
Main loop flow rate	On-line, computer logged	10/h
Letdown oxygen	On-line, computer logged	10/h
Letdown hydrogen*	On-line, computer logged	10/h
Inlet oxygen**	Periodic	1/d or as needed
Inlet hydrogen**	Periodic	1/d or as needed
Letdown conductivity	On-line, computer logged	10/h
Inlet conductivity	On-line, computer logged	10/h
Boron concentration***	Batch sample	1/2d or as needed
Lithium concentration***	Batch sample	1/2d or as needed
Letdown coolant activities***	Batch sample	1/2d or as needed

*Subject to availability of instrumentation. Note that the charging tank gas mixture pressure and temperature is monitored and hydrogen gas concentration can therefore be calculated.

**Inlet conditions are monitored by switching the letdown instrumentation to the inlet flow. This is normally done once per day but more frequently if abnormal conditions are suspected.

***Batch sample chemistry parameters are measured more frequently during start up or if abnormal chemistry is suspected. In general, the on-line conductivity measurements provides a good indicator of stable chemistry.

Note that a variety of other parameters that are not of direct importance to the experimental conditions are monitored for facility diagnostic purposes. These include such things as heater power level, gas consumption rates and letdown flow rate. These parameters are normally logged once per day.

4. Instrument Calibration Procedures and Safety Plan

In preparation.

Figure 1. Cross-section of the MITR-II giving the general layout of the in-core corrosion loop

Figure 2. Cutaway view of the in-core autoclave showing two pairs of CFCC specimens with spacers and flow shroud

Figure 3. Schematic illustration of the primary flow loop

Figure 4. Schematic diagram of the coolant chemistry and pressure control system