Quarterly Progress Report on Advanced
Gas Cooled Nuclear Reactor Materials
Evaluation ^{and Development} Program for the Period
April 1, 1977 Through June 30, 1977

MASTER

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States ERDA, nor any of their employees, nor any of their contractors, subcontractor, or their employees, make 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

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, 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. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. 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.

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
Foreword	i
Abstract	ii
1.0 Introduction	1
2.0 Program Status	4
2.1 Task 1: Selection of Candidate Alloys	4
2.2 Task 2: Materials Procurement and Characterization	10
2.3 Task 3: Testing and Analysis Facilities and Equipment	11
2.4 Task 4: Simulated Reactor Helium Environment Facilities & Equipment	18
2.5 Task 9: Data Management and Program Management	32
3.0 Work To Be Completed During the Next Quarter	37

LIST OF TABLES

<u>Table</u>	<u>Page</u>
2.1 Selected Alloys and Coatings for DCHT	6
2.2 Nominal Compositions (w/o) of Alloys for DCHT Screening Program	7
2.3 Nuclear Process Heat Alloys and Coatings	8
2.4 Nominal Compositions (w/o) of Alloys for NPH Screening Program	9
2.5 Console Gas Chromatographs	27
2.6 Candidates for the Portable Gas Chromatograph	29

LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
2.1 General View of the Helium Environment Materials Testing Laboratory	13
2.2 Layout Drawing of the Water Pump/Heat Exchanger Designed to Provide Demineralized Cooling Water to the Laboratory.	15
2.3 Assembly Drawing of the Lower End of a Ceramic Retort Tube for the Multiple Specimen Screening Creep Equipment Showing the Clamping Ring (arrow) for Attaching the End Fitting.	17
2.4 Advanced Gas Cooled Nuclear Materials Evaluation and Development Program.	34

FOREWORD

The Advanced Gas Cooled Nuclear Reactor Materials Evaluation and Development Program is being conducted for the Division of Nuclear Research and Applications, U.S. Energy Research and Development Administration by the Energy Systems Programs Department, General Electric Company, Schenectady, New York, 12345. The Program is under the direction of Mr. J. E. Fox, ERDA Program Manager, and is being managed for the General Electric Company by Mr. R. G. Frank, Manager, Materials Technology. The Principal Investigator for the Program is Dr. O. F. Kimball, Manager, Gas Reactor Materials with responsibility for overall technical coordination. Other major contributors to the Program are:

Dr. R. V. Hillery, Aircraft Engine Business Group,
Cincinnati, Ohio

Project Manager - Materials for Direct Cycle Helium
Turbine (DCHT); Program Data Analysis -
(Mr. G. Best, Dr. R. Hand)

Dr. R. M. Goldhoff, Large Steam Turbine-Generator Operation,
Schenectady, New York

Project Manager - Materials for Nuclear Process Heat
Systems (VHTR/NPH) - (Mr. S. Hayden)

Mr. C. L. Ehde, Advanced Energy Programs, Cincinnati, Ohio
Project Engineer - Simulated Reactor Helium Gas Supply
System

Mr. R. W. Stepien, Large Steam Turbine-Generator Operation,
Schenectady, New York

Project Manager - Materials Testing in Simulated Reactor
Helium (Dr. D. Baldwin, Mr. R. Elton)

Dr. A. W. Urquhart, Corporate Research and Development Center,
Schenectady, New York

Project Manager - Project Support for Simulated Reactor
Helium Gas Chemistry (Dr. H. S. Spacil) and
Corrosion (Dr. D. W. McKee).

ABSTRACT

This report presents the results of work performed from April 1, 1977 through June 30, 1977 on the Advanced Gas Cooled Nuclear Reactor Materials Evaluation and Development Program. The objectives of this program are to evaluate candidate alloys for Very High Temperature Reactor (VHTR) Process Heat and Direct Cycle Helium Turbine (DCHT) applications, in terms of the affect of simulated reactor primary coolant (impure Helium), high temperatures, and long time exposures, on the mechanical properties and structural and surface stability of selected candidate alloys. A second objective is to select and recommend materials for future test facilities and more extensive qualification programs.

Work covered in this report includes completion of alloy selection for the screening tests; the alloys selected for potential VHTR Nuclear Process Heat (NPH) applications, and for potential DCHT applications are listed. The present status on the simulated reactor helium loop design and construction, and the design and construction progress on the testing and analysis facilities and equipment are discussed.

Section 1.0

INTRODUCTION

ADVANCED GAS COOLED NUCLEAR REACTOR
MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

1.0 INTRODUCTION

The Very High Temperature Reactor (VHTR) Process Heat and the Direct Cycle Helium Turbine (DCHT) Systems are being developed to provide an expanded nuclear power capability. In both cases the use of high temperature helium-cooled reactors will require a knowledge of the long term properties of reactor fluid containment materials and of the materials used in constructing related hardware. Because of the very high temperatures required for process heat and DCHT applications, the need is evident for materials testing in the proper helium environment in the temperature range from 960 - 1093°C (1400 - 2000°F) on those alloys which might serve as materials of construction for heat exchangers, ducting liners, turbine buckets and vanes and other high temperature structural components.

The objectives of the Advanced Gas Cooled Nuclear Reactor Materials Evaluation and Development Program are to:

1. Evaluate candidate alloys for Very High Temperature Reactor (VHTR) Process Heat and Direct Cycle Helium Turbine (DCHT) applications, in terms of the effect of the primary coolant exposure and thermal exposure on:

- o Surface stability (corrosion resistance) in 10,000 hours.
- o Structural stability in 10,000 hours - changes in micro-structure and tensile, impact and fatigue properties.
- o Creep properties in 15,000 hours.
- o Stress rupture properties in 10,000 hours.
- o Fatigue properties.

Low cycle 10^3 to 10^5 cycle

High cycle 10^5 to 10^7 cycle

2. Select and recommend materials for future test facilities and more extensive qualification programs.

The data base will permit the selection of materials of construction and should provide a reliable level of design data for the construction of various items of development hardware leading to the successful development of VHTR and DCHT systems. The present lack of an accurate measure of materials capabilities under these high temperature helium environments is a factor which is currently limiting the development of the VHTR/DCHT systems. This program is intended to provide such initial information.

Section 2.0

PROGRAM STATUS

2.0 PROGRAM STATUS

The Advanced Gas Cooled Reactor Materials Evaluation and Development Program is organized into nine (9) tasks which are designed to accomplish the program objectives listed in Section 1.0 of this report. The task structures (Program Work Breakdown Structure) and a brief description of each task (Program Work Statement) have been included in the first quarterly report: C00-2975-3.

During the third quarter of the program, work was completed on Task 1 (Selection of Candidate Alloys); a final Alloy Selection Report will be issued during the fourth quarter of FY1977. Work has continued on Task 2 (Materials Procurement and Characterization), Task 3 (Testing and Analysis Facilities and Equipment), Task 4 (Simulated Reactor Helium Environment Facilities and Equipment), and Task 9 (Data Management and Program Management).

2.1 TASK 1 - SELECTION OF CANDIDATE ALLOYS

The final selection of candidate alloys for the Nuclear Process Heat (NPH) and Direct Cycle Helium Turbine (DCHT) screening programs was resolved May 24, 1977 in a meeting held at ERDA (Germantown, Maryland). Representatives from ERDA, General Atomic Co. (GAC), and General Electric Co. (GE), discussed GE's recommendations, as given in a preliminary draft, C00-2975-7, of an alloy selection report ("Selection of Alloys for the Advanced Gas Cooled Nuclear Reactor Materials Evaluation and Development Program) and the comments received from reviews of that report. The report was reviewed by materials engineers from the Battelle Columbus Laboratories, Stellite Division of Cabot Corp., General Atomic Co., International Nickel Co., NASA (Lewis Research Center), Oak Ridge National Lab., and Westinghouse Electric Co. The final selection of candidate alloys listed in the following sections of the report, have been approved by the ERDA Program Manager.

Input from discussions with personnel from Kernforschungsanlage (Julich, FRG) and the High Temperature Materials Program (Wimborne, England) also was of considerable value in making the final decisions on the alloys for the screening program.

2.1.1 Direct Cycle Helium Turbine (DCHT) Alloy Selection - R. V. Hillery

Based on input from many government and industry technical personnel, the recommended list of DCHT materials for helium loop testing has been finalized. The general considerations in alloy selection and the selection approach discussed in the last Quarterly Report were followed throughout and the final list (see Table 2.1) is essentially the same as that proposed earlier this year. Data generated recently by both General Atomic and HTMP have resulted in the addition of IN-738 and one of the following alloys: NX-188, WAZ-16, TRW VIA, Illium W, TAZ-8B, and the reduction in emphasis on YD-Ni, YD-NiCrAl, M-21, and MM-004. These latter four alloys will now be tested only in the unstressed condition to measure corrosion/thermal stability effects. Nimonic 115 was added to include a wrought Ni-base superalloy in the DCHT testing to evaluate the effect of different product forms. It also should be noted that the two ducting alloys, Inconel 617 and Hastelloy X, although not appearing in Table 2.1, are included with the NPH alloys and the data generated will suffice for both systems, since all screening tests will be done in one gas environment.

The chemical compositions of the alloys to be evaluated for DCHT application are listed in Table 2.2.

2.1.2 Nuclear Process Heat (NPH) Alloy Selection - S.Z. Hayden & R.M. Goldhoff

Alloys for potential process heat applications, selected for the screening program, are listed in Table 2.3. Alloys were selected from the wrought, cast and oxide dispersion strengthened classes of alloys on the basis of tensile, yield, and creep-rupture strength, metallurgical stability and embrittlement resistance, corrosion resistance, fabricability and availability.

TABLE 2.1
SELECTED ALLOYS AND COATINGS FOR DCHT

<u>Alloy</u>	<u>Condition</u>	<u>Alloy Type</u>
Mo TZM -Coated	UC* & C	Mo-Base Alloy
YD-Ni*		Oxide Dispersion Strengthened Nickel-
MA-754		Base Alloy
YD-NiCrAl*	UC* & PO*	↓
MM-247		Cast Nickel-Base
MM-247 DS		Superalloy, γ' Strengthened
Rene' 80	UC & PO*	↓
Rene' 100	UC & PO*	
M-21*	UC* & PO*	
IN-713LC	UC, PO*, C*, C+PO*	
MM-004*		
IN-738		↓
Nimonic 115		Wrought Nickel-Base Superalloy, γ' Strengthened
**NX-188, WAZ-16, TRW-VIA, Illium W, or TAZ-8B		Cast Nickel-Base Alloys Refractory Metal Strengthened

Code: UC: uncoated
C: Coated
PO: Preoxidized

* Unstressed

** One of this type of alloy

TABLE 2.2

NOMINAL COMPOSITIONS (W/O) OF ALLOYS
FOR DCHT SCREENING PROGRAM

<u>Alloy</u>	<u>Composition, W/O</u>										
	<u>Ni</u>	<u>Cr</u>	<u>Al</u>	<u>Ti</u>	<u>Co</u>	<u>Ta</u>	<u>Mo</u>	<u>W</u>	<u>Cb</u>	<u>C</u>	<u>Other</u>
Mo-TZM	--	--	--	0.45	--	--	Bal	--	--	0.015	0.09 Zr
YD-Ni	Bal	--	--	--	--	--	--	--	--	--	2Y ₂ O ₃
MA-754	Bal	20.5	.3	.5	--	--	--	--	--	0.05	0.6Y ₂ O ₃ , 1Fe
YD-NiCrAl	Bal	16	4.0	--	--	--	--	--	--	--	1.0Y ₂ O ₃
MM-247	Bal	8.3	5.5	1.0	10.0	3.0	0.7	10.0	--	0.16	1.5 H _f
Rene' 80	Bal	14.0	3.0	5.0	9.5	--	4.0	4.0	--	0.17	
Rene' 100	Bal	9.5	5.5	4.2	15.0	--	3.0	--	--	0.18	
M-21	Bal	5.7	6.0	--	--	--	2.0	11.0	1.5	0.08	
IN-713LC	Bal	12.0	5.9	0.6	--	--	4.5	--	2.0	0.05	
MM-004	Bal	12.0	5.9	0.6	--	--	4.5	--	--	0.05	1.3 H _f
IN-738	Bal	16.0	3.4	3.4	8.5	1.7	1.75	2.6	0.9	0.17	
Nimonic 115	Bal	15	5	4	15	--	--	--	--	≤0.2	
NX-188 ^(a)	Bal	--	8.0	--	--	--	18.0	--	--	0.04	
WAZ-16 ^(a)	Bal	--	7	--	--	--	2.	16.	2	0.18	.5Zr
TRW-VIA ^(a)	Bal	6.1	5.4	1.0	7.5	9.0	2.0	5.5	0.5	0.13	0.13Zr
Illium W ^(a)	Bal	16	--	--	--	--	17	4	--	0.08	6 Fe
TAZ-8B ^(a)	Bal	6.0	6.0	--	5.0	8.0	4.0	4.0	1.5	0.125	.004B, 1.0Zr

(a) One alloy from these five to be selected.

TABLE 2.3

NUCLEAR PROCESS HEAT ALLOYS AND COATINGS

<u>Alloy</u>	<u>Condition</u>	<u>Alloy Type</u>
Incoloy 800H	UC & C*	Wrought Iron Base Austenitic
Incoloy 802	UC	" " "
Incoloy 807	UC	" " "
Incoloy DS	UC	" " "
HD-556	UC	" " "
Manaurite 36XS	UC	Cast Iron-Base Austenitic
Manaurite 900*		" " "
HK-40	UC	" " "
IN-519	UC	" " "
Hastelloy X	UC & C*	Wrought Nickel-Base Austenitic
Inconel 617	UC & C	" " "
Inconel 618E*	UC*	" " "
Inconel 625**		" " "
Mo-Re 2	UC	Cast Nickel-Base Austenitic
IN-643	UC	" " "
MA-956E	UC	Oxide Dispersion Strengthened Iron Base Alloy

Code: UC - Uncoated

C - Coated

* - Unstressed exposures only

** - To be reconsidered for application above 1400°F

TABLE 2.4

NOMINAL COMPOSITIONS (W/O) OF ALLOYS FOR NPH SCREENING PROGRAM

Alloy	Composition, W/O										
	C	Ni	Co	Cr	Fe	Al	Ti	Mo	W	Cb	Other
Incoloy 800H	0.08	32.5	--	21.0	Bal	0.4	0.4	--	--	--	--
Incoloy 802	0.35	32.5	--	21.0	Bal	0.6	0.75	--	--	--	--
Incoloy 807	0.08	40.0	8.0	20.3	Bal	0.4	0.4	--	5.0	1.0	--
Incoloy DS	0.05	37.0	--	18.0	Bal	--	--	--	--	--	2.3 Si
HD-556	0.10	20.0	20.0	22.0	Bal	0.3	--	3.0	2.5	0.1	1.3 Mn, 0.9 Ta
Manaurite 36XS	0.40	34	--	26	Bal	--	--	--	2.0	1.5	2.0 Si
Manaurite 900	0.15	32.0	--	21.0	Bal	--	--	0.3	--	1.5	1.0 Si
HK-40	0.40	20.0	--	24.0	Bal	--	--	--	--	--	1.8 Si
IN-519	0.30	24.0	--	24.0	Bal	--	--	--	--	1.5	1.0 Si
Hastelloy X	0.10	Bal	1.5	22.0	18.0	--	--	9.0	0.6	--	1.0 Si
Inconel 617	0.07	Bal	12.5	22.0	--	1.0	--	9.0	--	--	--
Inconel 618E		54	≤0.1	23	Bal	--	--	--	6	--	--
Inconel 625	0.05	Bal	--	22.0	3.0	0.2	0.2	9.0	--	4.0	--
Mo-Re 2	0.4	51	--	33	--	1.0	--	--	15	--	--
IN-643	0.5	Bal	12.0	25.0	3.0	--	--	0.5	9.0	2.0	<.25Zr
MA-956E	--	--	--	20	Bal	45	0.5	--	--	--	0.5 Y ₂ O ₃

Because of the severe carburization observed in the GAC/HTMP tests on wrought nickel or iron-base solid solution strengthened alloys tests on coated materials will be given more emphasis than originally planned. Inconel 617 will be tested in the uncoated and coated conditions in screening creep and structural stability tests. Structural stability studies of other coated alloys (Incoloy 800H and Hastelloy X) will be performed and coated Incoloy 800H specimens will be used as replacement screening creep specimens.

Analysis of creep-rupture data recently provided by Huntington has indicated that no benefit is derived from Inconel 625 compared to Incoloy 800H or Incoloy DS in the 1800°F temperature regime. Additionally, this alloy also has been reported to lose ductility on exposure in the 650°C - 870°C (1200-1600°F) temperature range and therefore, is not recommended for evaluation in the current program.

Since the meeting at ERDA on May 24, 1977, it was found that IN-643 is being reformulated at the International Nickel European Research and Development Center. Therefore a replacement is being considered for this alloy. From work performed at Battelle Columbus Laboratories (BCL), Supertherm or possibly a derivative of HP-50 (35Ni-26Cr) containing either 5 percent tungsten or 5 percent tungsten plus 0.25 percent zirconium (designated HP-50WZ) were suggested. Information on the latter alloy will be obtained; if the data are encouraging HP-50WZ will be substituted for IN-643.

The chemical compositions of the alloys to be evaluated for NPH applications are listed in Table 2.4.

2.2 TASK 2 - MATERIALS PROCUREMENT AND CHARACTERIZATION

2.2.1 NPH Alloys - S. Z. Hayden, R. M. Goldhoff

A series of meetings was held with potential vendors to discuss the specification and procurement process for a number of the NPH program alloys.

Specifications for six (6) alloys (Incoloy 800H, Incoloy 802, Inconel 617, Incoloy 807, Incoloy DS and MA-956E) have been prepared and issued. Purchase orders have been issued for Incoloy 800H, Incoloy 802, and Inconel 617. Approximately 300 pounds of each alloy have or will be ordered; this amount is about twice that required for the present test program, will allow use of the same heat of material for the intensive program as will be used in the screening program, and will provide a material surplus for archive material or for possible future program modifications requiring additional material.

2.2.2 DCHT Alloys - R. V. Hillery

Many of the alloys on the list of selected materials for the DCHT program are alloys for which GE Specifications exist and need modification only to the extent that certain requirements of the existing specification are not appropriate or required for the current program. Other alloys on the list are sufficiently similar in basic requirements that a revision to an existing GE or ASTM Specification for another alloy can be made to satisfy the program needs. Two alloys, Mo-TZM and M-21, require preparation of new specifications and vendor contacts to assure that the requirements felt necessary for this program can be achieved. Work is proceeding in this Task and preparation of specifications for internal review and approval is in progress.

2.3 TASK 3 - TESTING AND ANALYSIS FACILITIES AND EQUIPMENT - D. Baldwin & R. Stepien

2.3.1 High Temperature Reactor Materials Testing Laboratory (HTR/MTL)

Construction of the (HTR/MTL) laboratory was initiated and progressed in a satisfactory manner during the quarter covered by this report. Specifically:

- Partitions - Existing partitions have been removed or relocated as required to accommodate the equipment for the laboratory.

FIGURE 2.1 General view of the Helium Environment Materials Testing Laboratory under construction. Note overhead wireways in place. Utility frames for multi-specimen stands are in foreground; aging/corrosion next, and individual test stand frames in the background.

- Water Pump and Heat Exchanger Package: The preliminary design layout of this unit is shown in Figure 2.2. The basic elements are pumps (D), heat exchanger (C), and storage tank (A). The two pumps are all stainless steel and are driven by 3 hp motors. If pump No. 1 fails, pump No. 2 is automatically started and alarms are activated to warn the laboratory operator. If pump No. 2 fails to start or fails before pump No. 1 has been repaired, solenoid valves are activated to switch from closed loop demineralized water to a domestic cold water supply which is circulated once through the laboratory and dumped.

The entire pumping and heat exchange package is designed so that the demineralized water circulated throughout the laboratory comes in contact only with stainless steel or copper alloys. Thus, the pumps and storage tank are of stainless steel construction and the U-tubes of the heat exchanger are of copper.

Some revisions to the system have been initiated since the drawing shown in Figure 2.2. was made. However, these have to do primarily with the physical arrangement of the various components and do not alter the basic functional concepts. The final design has been approved by GE engineers and construction of the unit begun.

2.3.2 Multi-Specimen Screening Creep Equipment

A design review by the vendor, Applied Test Systems, Inc. (ATS) for GE personnel was held in Schenectady on May 23, 1977. The final design approved by GE is in most respects identical with that described in the preceding quarterly report, except for the method of attachment of the retort end fittings to ceramic retort tubes. For this purpose a split, segmented clamping ring has been designed. This will be clamped to the retort tube near its end to serve as an anchor to which retorts and fittings are bolted. A thin layer of silicone sheeting is placed between the aluminum clamping ring and the ceramic retort tube to assure uniform distribution of the clamping force. This method of attachment has

FIGURE 2.2 Layout drawing of the water pump/heat exchanger designed to provide demineralized cooling water to the laboratory.

been tested full scale by ATS under conditions more severe than those called out in the GE engineering specification. The general arrangement of components is shown in Figure 2.3.

Layout drawings furnished by ATS have been reviewed and approved by the ERDA Program Manager.

ATS has completed the detail design and has begun prototype construction. Completion date for this first unit is mid-July, and it is scheduled for demonstration to GE personnel on July 26, 1977 at the ATS facility in Saxonburg, Pa.

2.3.3 Aging/Corrosion (Structural Stability) Equipment

Satec Systems, Inc. (SSI) has been identified as the preferred vendor for eight aging/corrosion stability stands. Again, the design is essentially identical to that described in the preceding quarterly report, except for end fittings to the retort tubes. In this instance, a system based on ceramic "protrusions" provided as an integral part of the retort tube is proposed. These will serve as anchors for an appropriate clip to retain the end assemblies. It is to be noted that negotiations with SSI have not progressed to the point of prototype construction. Purchase of the furnaces and stands has been initiated.

2.3.4 Single Specimen Creep-Rupture Equipment

ATS has been selected as the supplier of the 35 creep units and 10 stress-rupture units. Purchase of the furnaces, controllers, and test frames, has been initiated. Purchase of retorts, extensometry, and load train components awaits ERDA approval.

2.3.5 Test Temperature Control System

Research, Inc. (RI), suppliers of the integrated temperature control systems, has provided GE with technical information on the thermocouple reference junction boxes to be provided by RI and to be installed and wired

FIGURE 2.3 Assembly drawing of the lower end of a ceramic retort tube for the Multiple Specimen Screening Creep Equipment showing the clamping ring (arrow) for attaching the end fitting.

by GE. The locations of these two boxes have been fixed, and RI is currently pre-fabricating ribbon cables to connect them with the control console. GE also has furnished RI with cable length requirements for connection of their analog control signal outputs to the solid state power relays which will be located at the test stands. Software design is nearly complete. A design review with GE personnel is tentatively scheduled for mid-July. Delivery is still expected during September, 1977.

2.4 TASK 4 - SIMULATED REACTOR HELIUM ENVIRONMENT FACILITIES & EQUIPMENT

2.4.1 Simulated Reactor Helium "Loop" System Design and Construction - C.L. Ehde

2.4.1.1 Design

The quarterly report of April 1977 describes the general design of the HTR Materials Testing Laboratory. During this report period, some changes in the design have been made and are discussed. Also, the control systems have been further defined and are presented. The remainder of this section deals with the status of fabrication of system components and equipment procurement.

The general arrangement of the Laboratory remains essentially the same. Some changes to the cylinder gas manifolds have been made. Backup He gas (He with 100 ppm H_2) manifolds have been designed to handle 16, 1A, size cylinders which will supply the gas to both DCHT and VHTR/NPH loops in the event of a loop failure. The backup gas will pass through dual beds of Pt-Pd operating at 150°C (in parallel) and then through a single molecular sieve before entering the distribution system. The Pt-Pd bed will remove O_2 to very low levels and the molecular sieves will remove any H_2O vapor. Automatic control of backup gas remains a function of high O_2 level or low loop pressure. These parameters will be annunciated visually and audibly on the control console.

The 16 port gas analysis valve has been modified to include a shaft purge seal. This will prevent the in-leakage of air via back diffusion through the valve shaft seal.

The purge gas distribution system has been changed from copper to Type 304SS tubing. The purge discharge or dump piping system remains copper. The purge gas manifold which services all test stands has been reduced from a 20 cylinder to a 4 cylinder manifold.

A specification for cleaning stainless steel components for contact with the He gas was prepared and this cleaning specification added to all drawings covering fabrication of He containing components. A welding specification covering GTA welding of stainless steel components also was prepared and included on all appropriate drawings. Helium gas rather than Argon backup gas will be used on all welds. This will reduce the possibility of entrapping gas (argon) in the weld areas which might migrate into the He gas stream at a later time.

The control system for the He loops is outlined below:

Pumps - Manual individual start and stop. Three pumps normally in operation; automatic start of the fourth (standby unit) pump if the discharge pressure of any one of the three operating pumps falls below a preset level. Pressure switch sensor adjustable 0-50 psig. All pumps shutdown when the backup gas system is energized. Pump operation indicated by a green light on the graphic panel. Annunciation is by visual and audio signal indicating which pump has low pressure output and has shut down.

Purification Train - High H_2O Content - The Shaw Company Dewpoint Indicator is equipped with a contactor which is closed when H_2O level exceeds an adjustable preset level. This contact, in turn, energizes a time-delay relay which will open valves allowing a predetermined amount of He to pass through the $Mg(ClO_4)_2$ bed. The flow rate and time must be determined from operating experience. Both are adjustable within ranges. Only sensor #1 (in the main helium loop on the inlet side of the retorts) has high-low controls. An arming switch must be energized before the above automatic feature is available.

Low H_2O Content - The Shaw Company Dewpoint Indicator contact will close on low H_2O level. This contact will, in turn, energize a time-delay relay which will open valves allowing a predetermined flow of He to pass

through the H₂O injector. The flow rate and time must be determined from experience. Both are adjustable within ranges. A manual arming switch must be energized before this control feature is available.

High or Low H₂O Content - The water vapor content (dewpoint) of the He gas as sensed by the gas analysis system dewpoint sensor (sensor #2) is continually recorded. This single pen recorder is equipped with high and low contacts which can be adjusted and set at levels equal to or above or below the contact setting in the Shaw Indicator. They will be used to annunciate either a high or low dewpoint and will be adjusted to indicate to the operator that high or low limits are being approached which will require manual or automatic adjustment to the purification train.

Provisions for installing the #1 dewpoint sensor directly downstream of the H₂O injector are also included in this purification system piping design. Automatic operation of train valves with the sensor in this position is not contemplated and the primary purpose of a sensor in this position will be to get a feel for flow rate effects on dewpoint vs. oxalic acid temperature.

Further automation of the purification train is not contemplated initially; however, relays for automatic operation of the train solenoid valves will be provided in the consoles. The signals to operate these solenoids will come from a computer tie-in anticipated in the future.

All purification system solenoid valves are the normally closed type (i.e., power required to open). They can be individually manually operated from the graphic panel and a green light will indicate an open (energized) position. Each solenoid will be individually fused so that a shorted solenoid valve cannot disrupt control power to other components.

Backup He Gas - He backup gas can be automatically supplied to the main loop header in the event of high O₂ level or low pressure (i.e., pump failure). The solid electrolyte O₂ indicators are fed signals from two independent cells. One of these cells is located in the main supply

loop (sensor #1) and the second is located in the Gas Analysis System (sensor #2). Each cell is capable of tripping a high O_2 level contact in the indicator.

If the high level contact energized by sensor #1 closes, a relay in the console will be energized and the following actions will take place:

- The N.O. (normally open) main loop solenoid valve will close.
- The N.C. (normally closed) backup gas solenoid valve will open putting backup gas into the system.
- The loop dump valve on the inlet receiver tank will open, dumping the tank via a check valve and piping outside the building to atmosphere.
- The pumps (3) will be shut down.
- The inlet receiver tank fill solenoid valve will close.
- The annunciator will indicate "Backup Gas On" in visual and audible form.

Should the Hi O_2 level clear itself in some manner, the system will go back to its original mode with no manual operation required. However, the audible alarm will continue until it is acknowledged by manual means (i.e., acknowledge pushbutton depressed).

Backup gas also will be utilized if the supply side of loop pressure falls below a preset adjustable pressure. The action sensor in this case is a pressure switch in the supply header. The same action as outlined above for Hi O_2 level will result; however, the annunciator will indicate "Low Loop Pressure."

All of the above actions can be done manually and auto control will require that the operator arm the auto system with an arming switch located on the control console.

The second O_2 sensor (sensor #2), as previously stated, is in the Gas Analysis System. Should the contacts for the sensor be energized by a high O_2 level, an annunciator will be triggered. The trip point of this contact will be set at somewhat different level (amount of O_2 in the main He stream), so that the operator has some time to make manual adjustments to the system to reduce O_2 level thus avoiding the need to go on backup He gas. The above is true when the gas sampling scanner valve is in position #1 (main supply loop) or position #15 (return loop). Should the scanner valve be at any other position (say 2-14) automatic shutdown of isolation valves at the particular test stand or test stands being analyzed by this channel will occur isolating these stands from the rest of the system. The graphic panel will indicate via red lights which test stands have been isolated from the system. These valves will remain closed while the scanner valve continues to look at other sense line channels. The operator then must check out each of the test stands on this channel (maximum of 5 precision creep stands on one channel and 2 multi-string stands/channels by closing the sense header valve and opening the individual retort sense valves, one at a time, until he has located the offending retort. He can then isolate this particular stand from the loop, unlatch the relays holding the header valves closed, and the system is back in operation minus the offending retort. The worst case is where 5 stands are sensed by one sense line. Since the O_2 cell is a continuous reading device, the time required to locate the bad retort will be a function of the velocity of gas flowing from the retort to the gas analyzer system and the time required for the operator to open and close manual valves. The time is estimated at 15 to 20 minutes in the worst case where the offender is not found until samples from 4 stands have been analyzed.

Gas Analysis Scanning Valve - The Gas Analysis Scanner valve and its associated programmer and position indicator is a vital component in the Gas Analysis System. The rotary type valve contains 16 inlet ports, a common discharge port and a selected port. Gas from all 15 sense points

(Point #16 is a spare for bottle gas analysis) flow through the valve at all times. The vendor, Valco Instrument Company, of Houston, Texas, indicates a flow rate of approximately 300 cc/min per channel with a ΔP of 3 psi can be handled by the valve. Pressure drop available in the sense system will be approximately 5 psi so that the above flow rate may exceed the 300 cc/min (depending on the manual sense valve settings). During normal operation, 14 channels will be bypassed to a common discharge and the sensed channel will be directed to the gas analysis inlet manifold. Flow rate to the analysis manifold will be considerably higher (vendor indicates 1500 cc/min at ΔP of 3 psi) since the sensed discharge port only has to handle a single channel flow. Flow rate to the individual analysis sensors can be adjusted by the flow meter/valve ahead of each sensor.

The valve position (channel No.) is displayed in digital form on the indicator located in the center of the graphic panel. The Digital Valve Sequence Programmer controls the position of the rotary air driven valve. The programmer contains the electronic circuitry which allows the valve to operate automatically. Time intervals between positions can be pre-programmed over a broad range. Automatic full scan cycles can also be pre-programmed and valve positions can be skipped, if necessary, (i.e., no retorts on particular sense line). The programmer will also initiate scans and trigger recorder event markers.

General Controls - The inlet and discharge receiver tanks are equipped with pressure relief valves and also contain ports for connection of T/C Vacuum Gages which are used to monitor loop pumpdown during initial evacuation. An operating manual covering the general operating procedures will be prepared during the next reporting period. This will include schematics of the entire system which are not possible to include in this report because of drawing sizes.

2.4.1.2 Design Procurement and Construction Status

Design of the He loops is complete and all drawings have been issued. All major equipment, components, instrumentation, etc., are on order with

the exception of cylinder gas manifolds which are being quoted by three major gas supply companies. These bids are due before August 1, 1977. Fabrication of the He loop pumping, purification and instrumentation packages is underway at GE's AEP organization in Evendale, Ohio. A clean room has been set up and He piping subassemblies to be installed in the laboratory are under fabrication. Extensive cleaning facilities to insure a clean system have been set up at Evendale and all piping, fittings, etc., for the entire system are cleaned and bagged for use or shipment to Schenectady. Flow meters, gauges, valves, etc., are being assembled onto the graphic panel, pumps are being installed and package piping started. Instrumentation consoles are expected to arrive on site approximately 8/1/77 along with most of the units to be installed. The fabrication and cleaning of piping subassemblies to be shipped to Schenectady is being done in parallel with the pumping package buildup.

2.4.2 Simulated Reactor Helium Instrumentation and Control Systems - R.K. Elton

Instrumentation for the helium distribution and supply system has been extensively reviewed, and selections have been made. The major evaluations are discussed below.

2.4.2.1 Moisture Measurements

The candidates for trace moisture measurement include the following hygrometers: an EG&G Model 440 EXP, the Goldsmith hygrometer, a Panametrics Model 3000, and a Shaw Model SH2A. The EG&G model operates on the principle of optical detection of condensation on a chilled metal surface, the temperature of which is measured. This is the most accurate hygrometric technique available. However, the detection threshold is high, around -75°C (1.4 ppm V) which is approximately the same water concentration expected to be used in one of the loops. This factor, coupled with cost, cooling water requirements, and the lack of multi-probe capability outweighed the advantages of precision, and eliminated the EG&G model from further consideration. Goldsmith, a British firm, manufactures a hygrometer that monitors the electrolysis of H_3PO_4 to P_2O_5 . The current required to perform the electrolysis is a function of the water vapor concentration surrounding the probe.

Modifications of this probe by the HTMP staff to include a pulsed current mode indicate that accurate measurements at very low moisture levels are possible. While this instrument holds much promise for future use, it is still currently in the experimental stage, and would not prove satisfactory at this time for routine work. The Panametrics and Shaw models contain probes which consist of an $\text{Al}/\text{Al}_2\text{O}_3/\text{Au}$ capacitor, the capacitance of which is a function of the amount of water vapor which penetrates the porous Au coating. These probes are sensitive to moisture levels below 0.5 ppm V, and several may be interfaced to one instrument. Panametrics probes must be recalibrated annually at the factory for a fee. These probes are supplied with individual calibration curves which vary widely, making uniform centralized alarming difficult to achieve. However according to field tests, Shaw probes retain their calibration for several years, and the more uniform nature of the probe calibration curves facilitates accurate centralized alarming. Furthermore, the Shaw instrument is available with an expanded scale digital readout, and a 10 point calibration curve in the critical region between 0.2 and 1.0 ppm V, making this instrument the recommended method for the analysis and control of trace levels of water vapor. A two probe Shaw instrument, alarmed for high and low moisture levels was ordered. For the portable analysis cart, a single probe Shaw hygrometer with a dial readout was selected.

2.4.2.2 Console Gas Chromatograph

A gas chromatograph to be used on the loop console has been ordered. This instrument will analyze gas samples from various locations on the loop for H_2 , O_2 , N_2 , CO , CO_2 , and CH_4 , and is planned to eventually interface with a computer for automated control of the loop gas composition. Since the sensitivity of the instrument is usually limited by the detector, a number of different detectors were investigated. The helium ionization detector (HID) is generally accepted as being the most sensitive detector available for the analysis of a wide range of species, and is capable of detection near the parts per billion level. This device measures the current at a charged plate which results from the migration of ions formed

from the collision of gas molecules with metastable helium atoms. Virtually any gas that contacts the metastable helium will be ionized and detected. Two other detection systems, the electron capture detector (ECD) and the photoionization detector (PID) rival the HID in sensitivity, but are only applicable to specific types of gases. The ultrasonic detector (USD), which measures variations in the speed of sound, appears to be universally applicable to all types of gases, and can be made very sensitive. However, it is still being developed, and commercial models at competitive prices are not expected within the next year. The flame ionization detector (FID) operates on the same principle as the HID except that the ions are produced thermally in a flame. The FID, which can detect gases as dilute as 0.01 ppm V, is sensitive only to flammable substances, and thus requires a methane converter for the analysis for CO and CO₂. This converter does introduce some dead space into the system resulting in peak broadening and poorer resolution. The thermal conductivity detector (TCD) measures differences in the rate of heat transfer from a heated filament or thermistor, and is the least sensitive of the detection systems considered. The best TCD's generally cannot measure below 1 ppm V for any given species. However, the design simplicity and the ability to monitor most gases make it an attractive choice.

Based on the above instrumental limitations and the possibility of requiring the analysis of O₂, N₂ and CO₂ at concentrations lower than 1 ppm V, the instruments shown in Table 2.5 were given consideration. Instrument D required specialty construction and was dismissed on economic grounds. Both the E and A models require column switching facilities in order to direct the various components to the proper detector. This requires activation of a valve, which introduces the possibility of air inleakage. Column switching also causes sudden pressure changes in the system, resulting in probable baseline fluctuations in the output during sensitive measurements. Furthermore, reliance on a TCD/FID system would mean that analysis for N₂ would probably occur at or below the detection limit of the TCD. The choice of these instruments appears unwise, and they were ruled out.

TABLE 2.5
CONSOLE GAS CHROMATOGRAPHS

<u>Instrument</u>	<u>Location of Manufacturer</u>	<u>Column Switching</u>	<u>Detector(s)</u>	<u>-----Detection Limit (ppm V)-----</u>						<u>Cost Factor (Recorder Not Included)</u>
				<u>H₂</u>	<u>N₂</u>	<u>O₂</u>	<u>CO</u>	<u>CO₂</u>	<u>CH₄</u>	
A	U.S.	yes	TCD/FID	>50	5	5	0.05	0.05	0.05	1.8
B	Italy	yes	HID (1)	0.01 ^(a)	0.05	0.05	0.1	0.02	0.02	1.4
C	U.S.	no	HID (2)	0.05	0.01	0.01	0.02	0.02	0.01	1.2
D ^(b)	U.S.	no	HID (2)	0.08	0.05	0.05	0.05	0.02	0.01	5.0
E	U.S.	yes	TCD/FID	500	2	2	0.05	0.05	0.005	1.0

(a) Neon required as quenching gas.

(b) Detection limits for D inferred from instrument design.

Because of their HID systems, both the B and C models appeared promising. The B instrument has several features which indicate that it would be a risky choice. Besides column switching problems, the instrument requires neon in the carrier gas in order to detect hydrogen, which increases the operating expense. Since the producer is an Italian firm with no domestic users or service, extended down time in the event of a component failure could result. The C model incorporates 2 HID's with no column switching. The instrument is designed in a modular fashion to allow one column and detector to continue to operate in the event one component fails. This would allow continued analysis for at least 5 of the 6 components in the gas mixture. Replacements from the manufacturing plant can be acquired within a week.

Reports from three different users of the gas chromatograph indicated that the instrument is capable of continued routine analysis at 1 ppm V or less for long periods of time. Two of these instruments have been in use for over 5 years. One report indicated problems with arcing in the detector, but this was apparently due to faulty installation procedure on location. Model C (Antek 320D) was selected for the helium loop project.

2.4.2.3 Portable Gas Chromatograph

A gas chromatograph was chosen as part of the portable analysis cart. This instrument is needed to measure the composition of the gas inside a retort during purging, prior to the opening of the retort to the loop. Sensitive measurement of O_2 , N_2 , and CO_2 are required. The instruments investigated are given in Table 2.6. The instruments containing a flame ionization detector (FID) are capable of very sensitive analysis of carbonaceous species. However, the flame requires air in cylinders which would be difficult to mount on a cart that must negotiate some narrow places in the test area of the lab. Continuous use of the FID could require a new cylinder of air as often as once a week. Furthermore, use of an FID requires separate measurements of N_2 , O_2 and H_2 on a thermal conductivity detector (TCD), thus involving added costs to allow for column

TABLE 2.6

CANDIDATES FOR THE PORTABLE GAS CHROMATOGRAPH

<u>Instrument</u>	<u>Detector (s)</u>	<u>Number of Gas Cylinders Required</u>	<u>Detection Threshold</u>	<u>Cost Factor (Recorder) Not Included)</u>
A	TCD	1	2-3 ppm V N ₂ , O ₂ , CO ₂ , CH ₄ , CO 500 ppm V H ₂	1.0
B	TCD	2	3-4 ppm V N ₂ , O ₂ , CO ₂ , CH ₄ , CO 25 ppm V H ₂	1.5
C	TCD/FID	3	0.05 ppm V CO ₂ , CH ₄ , CO 3 ppm V N ₂ , O ₂ ; 500 ppm H ₂	1.5
D	TCD	1	1-2 ppm V N ₂ , O ₂ , CO ₂ , CH ₄ , CO 100 ppm V H ₂	1.5
E	TCD/FID	3	0.05 ppm V CO ₂ , CH ₄ , CO 1-2 ppm V N ₂ , O ₂ ; 100 ppm V H ₂	2.1
F	TCD	1	10 ppm V N ₂ , O ₂ , CO ₂ , CH ₄ , CO 1000 ppm V H ₂	>1.0
G	TCD	1	25 ppm V N ₂ , O ₂ , CO ₂ , CH ₄ , CO 500 ppm V H ₂	1.5
H	FID	3	0.05 ppm CH ₄ , CO ₂ , CO No analysis for H ₂ , N ₂ , O ₂	2.2

switching. An instrument with a highly sensitive TCD appears to be the best solution. Only two vendors, (A,B,C,) and (D,E), manufacture a TCD capable of sensing gases below the 10 ppm V level. Instrument E does not carry a performance guarantee, and is also more expensive. Instrument A (Carle III) is guaranteed to measure gases as dilute as 3 ppm V, and its modest price makes it the best choice.

2.4.2.4 Leak Detector

A helium leak detector, Veeco Model MS-9, provided by the General Electric Co., will be used for this program. This model has the reliability and sensitivity required for leak checking the "helium loops", retorts, etc.

2.4.2.5 Stainless Steel Cleaning Specifications

Specifications for the cleaning of all stainless steel parts to be incorporated into the helium loop have been issued. The specifications are designed to eliminate chemical contaminants such as oils, which could significantly alter the loop gas chemistry, and to remove foreign particles that could lodge in critical areas. The cleaning conditions are necessarily strict because even trace amounts of contaminants would be difficult or impossible to remove once the system is welded together.

The procedure involves a coarse removal of grease and oils in the tubing by repeated swabbing with lintless cloths soaked in an organic solvent. The tubes are then immersed in a standpipe filled with a degreasing solvent, and scrubbed thoroughly with stainless steel or nylon brushes. The tubes are rinsed with the same solvent, then with acetone. Drying is accomplished in the standpipe itself under flowing helium at 150°C. The standpipe will serve as a clean shipping and storing container. Small parts and fittings are to be cleaned ultrasonically, rinsed with acetone, and dried in an oven at 150°C. All work is to be done in a designated clean room, to be prepared according to specification.

Similar specifications have been issued for the cleaning of parts containing a brazed joint. This involves aqueous removal of the flux, rinsing with acetone, and drying. A separate specification has been drawn

up to outline the handling and storage of stainless steel materials to be used in the final loop assembly, and is intended to maintain the high level of cleanliness until all parts are secured into the system.

Arrangements have been made to clean all the stainless steel parts at one location. This will result in a considerable savings of time and money in the preparation and maintenance of the clean room. It eliminates a costly duplication of equipment. Furthermore, the cleaning technique will be more uniformly applied to all work.

2.4.2.6 Loop Gas Analysis Design Improvements

Several improvements have been made on the basic design of the helium loop. The location of the hygrometer probes to allow optimum control of the loop moisture level has been established. Two sensors are to be used, one in the gas analysis system, which will receive samples from 16 different locations in the loop, and one in the main header leading to the retorts. This latter probe will be alarmed, and eventually used to control the addition or removal of water in the loop. The probe in the sensing line will not be alarmed, and will serve to gather experimental data, such as depletion effects in the retorts. A probe holder will also be installed in the water injection line so that temporary measurements of the water vapor level in this line may be made for experimental purposes.

The 16 port gas sampling valve used to direct a stream of gas from a selected site on the loop to the gas analysis system will be housed and purged with helium. This feature reduces the possibility of air inleakage.

The bottled purge gas which will be flushed through the loop in the event of a pump failure or gross oxygen contamination will be scrubbed of oxygen by passage through two cannisters filled with Pt/Pd catalyst. The catalyst serves to produce water by allowing a reaction with hydrogen that is purposely blended into the gas mixture. The cannisters will be arranged in parallel, and the combined gas stream will be passed through a drying cannister.

The portable analysis cart is being equipped with an isolation valve at the inlet connection, and a bubbler on the exit line. These features allow the cart to be connected to the loop without contaminating either the loop or the instrumentation.

The possibility of computer automation of gas analysis by gas chromatography and hygrometry, and subsequent system control based on data interpretation is currently being investigated on two fronts. The staff at the UCLA chemistry department has developed a system for centralized computer handling of data from simultaneous experiments, one of which is gas chromatography. Their advice appears to be helpful. The vendor of the microcomputer used to control the system temperatures is also being considered for soft- and hardware. They suggest that it is possible to acquire and quantitate chromatographic data, compare this information to the desired gas composition, and, knowing system variables such as loop volume and circulation rates, send signals to the appropriate components on the purification and addition lines to make the necessary adjustments.

2.5 TASK 9 - DATA MANAGEMENT AND PROGRAM MANAGEMENT

2.5.1 Data Management

2.5.1.1 Data Acquisition

The test matrices for each material and property are being reviewed to assure a coordinated approach by the VHTR/NPH and DCHT materials test programs and to assure that the planned testing is compatible with evaluation and analysis requirements. Also, coincident with the material procurement phase and prior to the testing phase, the appropriate encoding forms are being selected.

- Data Encoding Coordination: Considerable progress has been made on the mechanics of data acquisition. Modified, revised and additional encoding forms are being developed. These forms will permit suitable

computer input to provide for management control of the test program.

- Encoding Instructions: Instructions are being written to ensure the proper encoding of information on the computer input forms. The forms and instructions required during the procurement phase should be ready for review by mid-July.

2.5.1.2 Data Storage and Retrieval

The definition of the material, material condition, test environment and the associated test data will be stored in a centralized computer data bank with the capability of selective retrieval. This data bank will be a revised or modified version of the current GE-AEBG data bank computer program.

The current GE-AEBG system has been reviewed for compatibility with the present program. No modification of concept or logic has been required. However, the system for this program will be more complex than the GE-AEBG system because of the time dependence required for program management. Progress is being made on the additional concepts and logic required by this characteristic of the system.

2.5.2 Program Management

A meeting was held with HTMP personnel at Wimborne, May 2-4, to review the latest results from Oslo Phase 4 GAC/HTMP testing of VHTR materials. Alloy selection for the ERDA/GE program was reviewed in light of recent HTMP/GAC results. The data had a definite effect on the final materials selection for this program.

In a meeting held at ERDA, Wash., D.C., representatives from ERDA, General Atomic Co., and General Electric, final decisions on alloy selection for the screening phase of the ERDA/GE program were made.

The Program Master Schedule is shown in Fig. 2.4.

Figure 2.4 Advanced Gas Cooled Nuclear Materials Evaluation and Development Program

Task 1 (Alloy Selection) has been completed except for revising the Alloy Selection Report. This report should be completed by the end of August.

Task 2 (Materials Procurement and Characterization) is about two months behind schedule in the Materials Procurement Cycle as a result of the delay in approval of the alloys for the program. This will result in one to two months slippage for completion of this task.

Task 3 (Testing and Analysis Facilities and Equipment) and Task 4 (Simulated Reactor He Environment Facilities and Equipment) are about three months behind schedule which will probably result in about two to three months slippage in the start of testing.

Section 3.0

WORK TO BE COMPLETED DURING THE NEXT QUARTER

3.0 WORK TO BE COMPLETED DURING THE NEXT QUARTER

1. Task 1 - Final Alloy Selection Report will be issued.
2. Task 2 - Finalize all materials purchasing specifications and order all materials.
 - Initiate characterization on as-received materials.
3. Task 3 - Complete purchasing cycle for screening creep and aging/corrosion equipment.
 - Complete check-out of prototype screening creep test unit.
4. Task 4 - Complete construction of utility frame helium distribution system for Loop 1.
5. Task 5,6,7 - Finalize test matrices and test plans.
6. Task 9 - Encoding forms for data storage will be prepared and reviewed for approval.