DR. 584

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ADVANCED GAS COOLED NUCLEAR REACTOR
MATERIALS EVALUATION AND DEVELOPMENT
PROGRAM

Progress Report, January 1—March 31, 1978

MASTER

June 26, 1978

Work Performed Under Contract No. EY-76-C-02-2975

General Electric Company
Energy Systems Programs Department
Schenectady, New York

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ADVANCED GAS COOLED NUCLEAR REACTOR MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

PROGRESS REPORT FOR PERIOD

JANUARY 1, 1978 — MARCH 31, 1978

**BY
PROJECT STAFF**

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**ENERGY SYSTEMS PROGRAMS DEPARTMENT
SCHENECTADY, NEW YORK 12345**

MASTER

JUNE 26, 1978

Prepared for
THE U.S. DEPARTMENT OF ENERGY
Under Contract No. EY-76-C-02-2975.A008

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FOREWARD

The Advanced Gas Cooled Nuclear Reactor Materials Evaluation and Development Program is being conducted for the Nuclear Power Development Division, U. S. Department of Energy, by the Energy Systems Programs Department, General Electric Company, Schenectady, New York 12345. The Program is under the direction of Mr. J. E. Fox, DOE 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:

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ABSTRACT

This report presents the results of work performed from January 1, 1978 through March 31, 1978 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 Nuclear Process Heat (NPH) and Direct Cycle Helium Turbine (DCHT) applications, in terms of the affect of simulated reactor primary coolant (Helium containing small amounts of various other gases), 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 the activities associated with the procurement of the materials for the screening test program, information from vendor certification for the materials received, and preliminary information from the materials characterization tests performed by General Electric. The construction status of the simulated reactor helium supply system, testing equipment, and gas chemistry analysis instrumentation and equipment are discussed. The final recommended impurity levels for the screening phase helium are presented and the rational behind this gas chemistry is discussed. Finally, the status of the data management system is presented.

ADVANCED GAS COOLED NUCLEAR REACTOR
MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

1.0 INTRODUCTION

The Very High Temperature Reactor (VHTR) Nuclear Process Heat (NPH) 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 primary coolant containment materials and of the materials used in constructing related hardware. Because of the very high temperatures required for Nuclear Process Heat (NPH) and DCHT applications, the need is evident for materials testing in the proper helium environment in the temperature range from 760 - 1093°C (1400 - 2000°F) on those alloys which might serve as materials of construction for heat exchangers, ducting, 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) Nuclear Process Heat (NPH) 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 NPH 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.

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.

2.1 TASK 1 - SELECTION OF CANDIDATE ALLOYS

The selection of alloys for the screening phase of the program has been completed. A list of the alloys selected for the program was presented in the Quarterly Report for the period July 1, 1977 - September 30, 1977: C00-2975-17.

2.2 TASK 2 - MATERIALS PROCUREMENT AND CHARACTERIZATION

2.2.1 Direct Cycle Helium Turbine (DCHT) Alloys - (E. W. Ross)

2.2.1.1 Materials Procurement

The status of the procurement of the DCHT alloys is presented in Table 2.1. All of the 12 selected alloys (13 alloy/process combinations: MM-0011 has been procured both in the conventionally cast and in the directionally solidified condition) have been received or are in transit.

2.2.1.2 Materials Characterization

o Chemical Analysis

The alloys received to date (Mo-TZM, MM-0011 CC and DS, Rene '80, Rene '100, IN 713LC, IN-738, NX-188, M-21 and YD-NiCrAl) have been specifically analyzed at General Electric for conformance

Table 2.1

DCHT BLADE & VANE MATERIALS

PROCUREMENT AND CHARACTERIZATION STATUS

<u>Alloy</u>	<u>Material Vendor</u>	<u>Status</u>
MM-0011 CC	MISCO	Received - In Machining
MM-0011 DS	TRW	Received - Machining to Begin 4/10/78
Rene' 80 CC	MISCO	Received - In Machining
Rene' 100 CC	MISCO	Received - In Q.A. Rupture and Tensile Test
IN-713 LC CC	MISCO	Received - In Q.A. Rupture and Tensile Test
IN-738 CC	MISCO	Received - In Machining
NX-188 CC	MISCO	Received - In Machining
M-21 CC	MISCO	Received - In Machining
YD-Ni	Special Metals	Not Received - Shipped 3/31/78
YD-NiCrAl	Special Metals	Received - Chemistry Incorrect ^(b) - New Materials to be Shipped 3/31/78
U-720	Special Metals	Not Received - Shipped 3/31/78
MA-754	Huntington Alloys	Not Received - Shipped 3/31/78
Mo-TZM	AMAX Special Metals	Received - In Q.A. ^(a) Rupture and Tensile Test

(a) Quality Assurance

(b) See Text for Details

to the specification and the vendor's chemistry certification. The analysis was primarily for the purpose of detecting any alloy mix-up (particularly a possibility in the cast alloys) or any element that was not specified or was at a far different level from the specification.

The quantity of bars analyzed for the cast alloys was equivalent to the number of molds cast for each alloy. The GE spectrographic analysis of the received material indicated the correct chemistry except for the YD-NiCrAl which was found to contain 5-6% cobalt. The YD-NiCrAl material was rejected and a replacement lot with essentially 0% cobalt has been shipped by the vendor. It is to be noted that the only YD-Ni available has been found by the vendor to have 0.9% cobalt. Although this level of cobalt is outside the desirable limits, it is not expected to have a significant effect on test results. The YD-Ni is being studied only in the unstressed condition, to allow comparisons of the environmental effects on ODS Ni, ODS Ni-Cr (MA-754) and ODS NiCrAl. Since YD-Ni is not used commercially, to obtain cobalt-free YD-Ni would be economically prohibitive. The vendor chemical analyses for the alloys received to date are shown in Table 2.2. All analyses are within the specifications requirements which are given in Table 2.3.

o Surface Grain Size

The surface grain size of the cast bars and pins received from Misco is shown in Table 2.4. The majority of the bars are within the 1.64 in. - 1/16 in. grain size aim. It is not believed that the grain size differences in any alloy will significantly affect the screening creep program.

o Optical Microscopy

As-received transverse sections of all of the alloys received to date plus sections of those alloys which were subsequently heat treated at General Electric (MM-0011 CC and DS, Rene'80, Rene'100, IN-738, NX-188) are in the process of being metallographically evaluated for conformance to each alloy specification.

Table 2.2

CHEMICAL COMPOSITION^(a) OF DCHT ALLOYS

Alloy	Heat No.	Cr	Ni	W	Fe	Co	Mo	Element, w/o		Cb	Ta	Zr	B	C	Other	NV _{3B}
								Al	Ti							
Rene' 80	7-11029	14.0	Bal	4.10	.09	9.30	3.9	3.00	5.00	-	-	.03	.01	.15	-	2.31
NX-188	6-99266	-	Bal	-	.14	-	18.00	7.90	-	-	-	-	-	.04	-	-
MM-0011 (CC+DS)	7-10768	8.20	Bal	9.80	.02	10.00	.64	5.45	.99	-	2.91	.07	.015	.14	1.41Hf	-
Rene' 100	7-11047	9.85	Bal	-	.20	15.18	3.03	5.46	4.19	-	-	.07	.015	.18	1.05V	2.46
IN-738	141C6869	15.82	Bal	2.50	.11	8.10	1.76	3.30	3.32	.65	1.60	.03	.011	.10	-	2.24
IN-713 LC	66B6965	12.00	Bal	-	.15	.10	4.10	5.85	.71	2.04	-	.06	.007	.06	-	-
TZM	TZM10834	-	<.001	-	.002	-	Bal	-	.43	-	-	.093	-	.022	-	-
M-21	B620	5.9	Bal	10.5	.18	.23	2.05	6.00	-	1.3	-	.14	.022	.10	-	-

(a) Values from vendor certifications.

Table 2.3

SPECIFIED CHEMISTRY RANGE FOR DCHT ALLOYS

Alloy	Specification Number	Cr	Ni	W	Fe	Co	Mo	Al	Ti	Cb	Ta	Zr	B	C	Other	NV _{3B}
Rene '80	EPD-01-0014-00-0	13.7-14.3	Bal	3.7-4.3	<.35	9.0-10.0	3.7-4.3	2.8-3.2	4.8-5.2	<.10	<.10	.02-.10	.01-.02	.15-.19		<2.32
NX-188	EPD-01-0017-00-0	--	Bal	--	<.35	<.10	17.5-18.5	7.75-8.25	--	--	--	--	--	.03-.05	Mn<.20 Si<.20 S<.015	--
MM-0011	EPD-01-0016-00-0	8.0-8.8	Bal	9.5-10.5	<.5	9.0-11.0	.5-.8	5.3-5.7	.9-1.2	--	2.8-3.3	.03-.08	.01-.02	.13-.17	1.2-1.6Hf	
Rene '100	EPD-01-0015-00-0	9.0-10.0	Bal	--	<1.0	14.0-16.0	2.7-3.3	5.3-5.7	4.0-4.4	--	--	.03-.09	.01-.02	.15-.20	0.90-1.10V	<2.46
IN-738	EPD-01-0019-00-0	15.7-16.3	Bal	2.4-2.8	--	8.0-9.0	1.5-2.0	3.2-3.7	3.2-3.7	0.6-1.1	1.5-2.0	.03-.08	.005-.015	.09-.13	--	<2.32
IN-731LC	AMS 5391B	12.0-14.0	Bal	--	<2.5	<1.0	3.8-5.2	5.5-6.5	.50-1.0	Cb+Ta 1.8-2.8	--	.05-.15	.005-.015	.04-.06	--	--
TZM	AMS 7819	--	--	--	<.02	--	>99.25	--	.40-.55	--	--	.06-.12	--	.010-.030	--	--
M-21	--	5.7	Bal	11.0	--	--	2.0	6.0	--	1.5	--	.12	0.02	.08-.15	--	--

Table 2.4

SURFACE GRAIN SIZE OF CAST BARS AND PINS OF DCHT ALLOYS

(Aim: 1/64 - 1/16 inch)

<u>Alloy</u>	<u>Test Bars (Gauge) (a)</u>	<u>Surface Stability Pins (b)</u>
MM-0011CC	1/64 - 1/8 ^(c)	1/64 - 1/8
Rene' 100	1/64 - 1/16	1/64 - 1/32
Rene' 80	1/64 - 1/16	1/64 ^(d)
IN-713 LC	1/64 - 1/8	1/32 - 1/16
IN-738	1/64 - 3/32	1/64 - 1/8
NX-188	1/32 - 3/32	1/32
M-21	1/16 - 1/4	1/32 - 1/8

(a) One bar/each 16 cast examined [seven to eight molds of 16 test bars each were cast for all alloys except M-21 (one mold)] .

(b) One bar/alloy examined (one mold of 36 pins/alloy was cast).

(c) One bar of eight examined to 1/8 in., balance 1/64 - 1/16 in.

(d) Will not be used in program since different Rene' 80 heat was cast; all Rene' 80 pins will therefore be machined from test bars.

o Machining

Metcut Research Associates, Cincinnati, Ohio was selected as the machining vendor. Metcut is the machining vendor for the Nuclear Process Heat (NPH) alloys and they have been machining stress-free test specimens for General Electric for over 20 years. They also manage the U. S. Government Machinability Data Center.

A purchase order and supporting documentation for DOE Contracting Officer approval for the sub-contract was prepared, submitted, and approved during this quarter.

A total of 257 tensile/rupture specimens are being machined by Metcut. These will be used for:

- 1) quality assurance testing of incoming material to the specification (or equivalent if there is no specification requirement) for all 13 alloy/processes,
- 2) 300-hour creep/rupture testing of 10 alloy/processes to provide assurance that the proper stresses were selected for the long time screening creep tests,
- 3) tensile specimens for the 10 alloy/process specimens which will be exposed (without stress) in the helium environment for up to 10,000 hours at 3 or 4 temperatures and tested after exposure.

Three alloys, M-21, YD-NI, and YD-NiCrAl, will not be creep/rupture or tensile tested other than the QC inspection of the incoming material. However, these three alloys will be part of the extensive surface and structural stability study program ("corrosion pins").

A total of 409 screening creep bars are to be machined for the long time helium/air tests. These will be from the same 10 alloy/process combinations previously noted.

A total of 376 surface stability pins will be machined from all 13 alloy/process combinations.

The cast tensile/rupture specimens and corrosion pins are heat treated (when required) at General Electric prior to all machining. The wrought material from large bars will be rough machined, heat treated (when required) at GE, and then final machined.

The machining of the quality assurance and the 300-hour creep/rupture bars have top priority. These are scheduled to be completed by late April to assure completion of testing prior to the beginning of the screening creep program at Schenectady.

After machining, all bars are being fluorescent penetrant inspected (FPI) prior to testing and the indications recorded if greater than 0.005 inch in size. (The cast bars have been radiographed by the casting vendor and the occasional defective bars scrapped.)

2.2.2 Nuclear Process Heat (NPH) Alloys - (S. Z. Hayden)

2.2.1 Materials Procurement

Mill products for twelve of the fourteen candidate alloys for the screening program have been received. Receipt of the final two alloys, in the form of centrifugally cast tubes, has been delayed by a strike at the vendor's plant.

2.2.2 Materials Characterization

o Machining

The DOE (Chicago) Contracting Officer approval to sub-contract machining of the nuclear process heat candidate alloy test specimens to Metcut Research Associates was received and a purchase order was placed. Characterization test specimen machining is well underway; two shipments of tensile and creep/rupture specimens have been received with additional deliveries expected by the end of April.

o Nondestructive Evaluation

Specimens machined from centrifugally cast tube and ODS bars are non-destructively evaluated by fluorescent penetrant inspection and radiography; samples fabricated from wrought plate are fluorescent penetrant inspected (the plate was previously inspected by ultrasonic techniques). The fluorescent penetrant inspection procedure is performed to the requirements of EPD-02-0014-00-0, Fluorescent Penetrant Inspection Engineering Specification. Radiographic inspection is performed in accordance with the requirements of Section III of the ASME Boiler and Pressure Vessel Code.

o Mechanical Properties

Characterization creep/rupture testing was initiated during this quarter. Specimens of Incoloy 802, Hastelloy X, and MA-956E are being tested under stresses that should cause rupture in about 300 hours.

Room temperature tensile properties have been determined for eight as-received NPH test materials. The results are presented in Table 2.5. Tests were conducted in accordance with the requirements of ASTM A 370, Standard Methods and Definitions for Mechanical Testing of Steel Products. An Instron Tensile Testing Machine was used with crosshead speed maintained at 0.0085 mm/sec (0.020-inches/minute) through the yield point. Crosshead speed was then increased to 0.085 mm/sec (0.200-inches/minute) for the remainder of the test. Where available, manufacturer's tensile data (Table 2.6) agrees well with the tensile results obtained by GE; for the samples tested, these results confirm the vendor's reported values that exceeded, in all cases, the minimum acceptable (specified) values.

2.2.2.3 Materials Joining Studies

Varestraint tests are being performed on the nuclear process heat alloys (except for MA-956E). The Varestraint Test is being performed to evaluate the tendency for hot cracking in the weld metal,

Table 2.5

AS-RECEIVED TENSILE PROPERTY DATA FOR NPH TEST MATERIALS

Alloy	Specimen Number	Condition For Tests	Temp. (°C)	0.2% Offset Yield Strength		Ultimate Tensile Strength		Elongation (% in 1 inch gage length)	Reduction -In- Area (%)
				MPa	(Ksi)	MPa	(Ksi)		
Incoloy 802	1100TN02	S.A. 1271-	R.T.	221	(32.0)	610	(88.5)	47.0	58.0
Incoloy 802	1100TN03	1282°C	R.T.	221	(32.0)	613	(88.9)	49.0	59.0
Incoloy 807	1200TN02	S.T. 1230°C/	R.T.	255	(37.0)	662	(96.1)	53.0	61.0
Incoloy 807	1200TN03	5 Min., WQ	R.T.	255	(37.0)	662	(96.0)	53.0	61.4
Incoloy DS	1300TN02	S.A. 1150°C/	R.T.	221	(32.0)	600	(87.0)	54.0	73.1
Incoloy DS	1300TN03	5 Min., AC	R.T.	221	(32.0)	600	(87.0)	53.0	73.1
Manuarite 900	1600TN03	As Cast	R.T.	190	(28.0)	541	(78.5)	42.0	43.4
Manaurite 900	1600TN04	As Cast	R.T.	190	(28.0)	543	(78.7)	39.0	43.4
Manaurite 36XS	1500TN02	As Cast	R.T.	269	(39.0)	569	(82.5)	15.3	15.4
Manaurite 36XS	1500TN03	As Cast	R.T.	268	(38.8)	585	(84.8)	16.0	16.2
Hastelloy X	1900TN02	S.T. 1177°C/	R.T.	358	(52.0)	776	(112.5)	49.0	61.0
Hastelloy X	1900TN03	31 Min., WQ	R.T.	357	(51.8)	772	(112.0)	49.0	60.5
Inconel 618E	1B00TN02	S.T. 1232°C/	R.T.	269	(39.0)	633	(91.8)	60.0	67.1
Inconel 618E	1B00TN03	1 hr., WQ	R.T.	248	(36.0)	633	(91.8)	63.0	73.1
MA-956E	1D00TN01	S.A. 1330°C/	R.T.	786	(114.0)	796	(115.5)	13.0	41.6
MA-956E	1D00TN02	1 hr., AC	R.T.	786	(114.0)	800	(116.0)	12.0	39.1

Table 2.6

VENDOR SUPPLIED QUALIFICATION PROPERTY DATA FOR NUCLEAR PROCESS HEAT TEST MATERIALS

Material	Applicable Specification	Condition For Qualification Tests	Stress Rupture Properties								Tensile Properties										Hardness (RB)	Grain Size ASTM #
			Temp. (°C)	Stress [MPa(ksi)]	Rupture Time		Elongation		Temp. (°C)	0.2% Offset Yield Strength		Ultimate Tensile Strength		Elongation		Reduction-in-area (%)						
					Actual (hr.)	Min. (hr.)	Actual (%)	Min. (%)		Actual [MPa (ksi)]	Min.(a) [MPa (ksi)]	Actual [MPa (ksi)]	Min.(a) [MPa (ksi)]	Actual (%)	Min. (%)							
Incoloy 800H	EPD 01-0001-00-0	S.A. 1232°C	---	---	---	--	---	--	20	186 (27.0)	170 (25)	520 (75.5)	450 (65)	55	30	69	67	1				
Incoloy 862	EPD 01-0002-00-0	S.A. 1271-1282°C	---	---	---	--	---	--	20	252 (36.5)	207 (30.0)	620 (90.0)	517 (75)	47	18	52	75	1				
Incoloy 807	EPD 01-0005-00-0	S.T. 1230°C/5 min., W.Q.	---	---	---	--	---	--	20	286(41.5)	250 (36.2)	666 (96.6)	620 (89.9)	53.1	30	--	86	3				
Incoloy DS	EPD 01-0004-00-0	S.T. 1150°C/5 min.-AC	---	---	---	--	---	--	20	227 (32.9)	170 (24.6)	600 (87.0)	480 (69.6)	51.1	30	--	86	0.0-5.5				
HD-55E	EPD-01-0008-00-1	S.T. 1177°C/30 min., W.Q.	816	131 (19.0)	129	24	149.0	25	20	379 (55.0)	324 (47.0)	793 (115.0)	689 (100.0)	57.0	40	70.0	--	3.5				
Manaurite 36XS	None	As-cast	---	---	---	--	---	--	20	310 (44.9)	245 (35.6)	534 (77.5)	441 (64.0)	15.5	8	--	--	--				
Manaurite 900	None	As-cast	---	---	---	--	---	--	20	205 (29.7)	186 (27.0)	499 (72.4)	441 (64.0)	42.5	25	--	--	--				
Manaurite 900	None	As-cast	---	---	---	--	---	--	20	194 (28.2)	---	475 (68.8)	---	44	---	--	--	--				
HK-40	EPD-01-0009-00-1	As-cast	982	41.4 (6.0)	Disc. 15.1	11	---	--	20	332 (48.2)	241 (35.0)	616 (89.3)	448 (65.0)	21	15	18.4	--	70/50 (c)				
Hastelloy X	EPD 01-0007-00-1	S.T. 1177°C/31 min., WQ	816	110 (16.0)	72	24	164	8	20	345 (50.0)	276 (40.0)	765 (111.0)	689 (100.0)	49.0	35	53.0	--	6				
Inconel 617	EPD 01-0003-00-0	S.A. 1204°C	---	---	---	--	---	--	20	317 (46.0)	207 (30.0)	755 (109.5)	620 (90.0)	75	35	56	84	00				
Inconel 618E	None	S.T. 1232°C/1 Hr., W.Q.	---	---	---	--	---	--	--	---	---	---	---	--	--	--	--	--				
MA-956E	EPD 01-0006-00-1	S.T. 1330°C 1 Hr., A.C.	1100	48 (7.0)	(b) 21	7.5	10	--	--	---	---	---	---	---	---	--	--	--				

Notes: S.A. - Solution Annealed
S.T. - Solution Treated

(a) Minimum Values are Specified Minima

(b) Step Loaded: 38 MPa/24 Hrs., 48 MPa/24 Hrs.,
59 MPa/5 Hrs.

(c) Columnarity: Hot End/Cold End, %

heat-affected zone, and base metal of an autogeneous welded specimen (gas tungsten arc welded). Six of the wrought process heat alloys have been evaluated by this method; preliminary results indicate that in order of decreasing resistance to hot cracking, the ranking of the alloys is as follows:

- o Inconel 617 (Best)
- o Incoloy 802
- o Incoloy 80/
- o Incoloy 800H
- o Hastelloy X
- o Incoloy DS (Worst)

During this quarter, a survey was conducted to obtain information about commercial welding practice (filler metals, welding methods, and welding procedures) for the eleven (11) NPH alloys that will be studied in the welded condition in the screening program (unstressed exposures). Decisions about weld metal and welding methods have been made and most of the weld wire required has been ordered. Table 2.7 lists the filler metals recommended by the alloy producers, the filler metals chosen, and the welding method to be used for each alloy.

The filler metals and covered electrodes chosen have been or are being ordered. The next activity planned in the materials joining program will be a subprogram to develop welding procedures required to produce sound welds with the weldmetals and weld methods selected. The geometries to be welded for wrought plate and centrifugally cast tube materials are shown in Figures 2.1 and 2.2, respectively. Test specimens for unstressed exposures will be machined from samples with sound welds by taking transverse sections from welded samples of flat plate of the geometry shown in Figure 2.1, longitudinal sections from welded samples of centrifugally cast tubes of the geometry of Figure 2.2. In all cases, test specimens for unstressed exposures will include weld metal, heat-affected zone, and base metal.

Table 2.7

SELECTED WELDING METHOD AND FILLER WIRES FOR WELDING STUDIES

Alloy	Vendor Recommendations		Weld Metal Chosen		Welding Method
	Filler Metal	Covered Electrode	Filler Metal	Covered Electrode	
Incoloy 800H	Inconel 82	Inco weld A	Inconel 80		Gas Tungsten Arc Welding (GTAW)
Incoloy 802	Inconel 625	Inconel 112		Inco Weld A	Gas Metal Arc Welding (GMAW)
Incoloy 802	Inconel 617				
Incoloy 807	Inconel 625	Inconel 112	Inconel 617		GTAW
Incoloy DS	NC 80/20	Inco weld A	NC 80/20		GTAW
HD-556	HD 556 Matching		HD 556		GTAW
HD-556	Composition				
Manaurite 36XS	Manaurite 36X		Manaurite 36XS		GTAW
Manaurite 36XS	Matching Composition				
HK-40	HK-40 Matching		HK-40		GTAW
HK-40	Composition				
IN-519	IN-519 Matching		IN-519		GTAW
IN-519	Composition				
Hastelloy X	Hastelloy X Matching		Hastelloy X		GTAW
	Composition				
Inconel 617	Inconel 617		Inconel 617		GTAW
	Matching Composition				
Mo-Re 2		22H		22H	GMAW

FIGURE 2.1 SCREENING WELD SPECIMEN (FLAT PLATE)

Notes:

1. All surfaces 63/ $\checkmark$, except ends. Ends not critical.
2. Scale: 1:4
3. Dimensions in inches (mm).

FIGURE 2.2 SCREENING WELD SPECIMEN (CENTRIFUGAL CAST TUBE)

2.3 TASK 3 - TESTING AND ANALYSIS FACILITIES AND EQUIPMENT - (D. H. BALDWIN)

2.3.1 High Temperature Reactor Materials Testing Laboratory (HTRMTL)

2.3.1.1 Water Distribution System (Equipment Cooling Water)

The water pump and heat exchanger package has been installed. Electrical connections are complete and include revision of vendor-installed wiring for compatability with the control/alarm system designed by GE. Cooling water connections to the heat exchanger have been made and tied into the laboratory supply and drain lines. Lines to the laboratory domestic cold water supply are installed but not connected. (Domestic cold water will be used only if both the primary pump and the standby pump fail; switching to domestic cold water is done automatically upon failure of the standby pump.)

Distribution supply and return lines to the laboratory area have been installed, along with feeders to the utility frames serving all multi-specimen creep (MS) stands, all structural stability (AC) stands, and the first ten single specimen creep/rupture (ST) stands. Additional valving has been installed at each of these test stand sites to accomodate the multiple supply/return ports of each individual test retort.

The system has been filled, flushed, and refilled with demineralized water. Proper operation of the primary pump and pressure-activated switchover to the stand-by pump have been verified. The few aspects of system checkout remaining await tie-in to domestic cold water supply. The system is now essentially complete and operational.

2.3.1.2 Electrical Systems

The power distribution panel serving the pumping, purification and gas analysis console for Loop No. 1 (PP&A-1) has been wired into the laboratory supply. Instrumentation wiring has been tied into isolation transformers. Wiring changes have been made within PP&A-1 to accommodate a revised control strategy. A switch box containing 13 switches has been installed. These serve to switch control of

utility-frame mounted solenoid valves from one PP&A to the other as test stands are switched from one gas chemistry to another.

Furnace control panels have been installed for all MS and AC test stands and for the first ten ST stands. These panels incorporate over-temperature controllers, time clocks, solid-state relays, thermocouple jacks, power relays, and indicating lights necessary for proper furnace operation. Panels for the MS stands were fabricated by Applied Test Systems; all others by GE. Power wiring into these panels and from them to the furnace heating elements has been installed, as have connections to the computer-based temperature control system (this includes thermocouple connections to the computer and solid state relay wiring from the computer for each power zone; MS and AC furnaces are five zones each, ST furnaces are three zones each).

The creep strain monitoring system for ST stands No.'s 1-10 has been installed. This includes wiring from each test stand site (2 LVDT's) to a channel scanner and connection of the scanner to a data logging unit located in the same cabinet as the temperature control system (the two systems are totally independent). Power supplies for the LVDT's have been installed in the ST 1-10 utility frame.

The power distribution system is complete and operational for the equipment needed to start the screening tests.

2.3.1.3 Helium Distribution System

The principal components of these systems are the two PP&A units. The first PP&A unit was received at the HTRMTL during this quarter; installation of the unit is nearly finished. Electrical modifications and connections are complete, and helium piping connections to overhead distribution piping and gas manifolds are essentially (90%) complete. Cooling water, air supply, helium exhaust, and water injector refrigeration remain to be connected before installation of this unit is actually complete. These items are comparatively simple, however, and have not been completed in order to facilitate leak checking operations.

Leak detection and repair for this unit has taken longer than expected and has resulted in delay in attaining operational status for the controlled purity helium system. It is now expected that operation of the loop in the pressurized mode will start approximately June 1, 1978; the commissioning program is expected to be completed by August, 1978.

Gas bottle racks and manifolds manufactured by Matheson have been received and installed behind the PP&A sites. Associated purification apparatus (nitrogen cooled charcoal traps for fill and purge gases; "Ridox" and molecular sieves for back-up gas) built by GE have been plumbed. All manifolds were cleaned to GE specifications after delivery. Piping connections from manifolds into PP&A-1 are currently being made and will be followed by leak checks.

Overhead helium distribution piping to the laboratory is complete. Connections to all utility frame subassemblies for MS, AC, and ST 1-10 test stands are complete. Remaining subassemblies for ST 11-45 are either missing vendor-delayed valving (ST 11-20 and ST 21-30 subassemblies) or still in fabrication (ST 31-35 and ST 36-45 subassemblies). These do not affect the screening portion of the test program.

2.3.2 Multi-Specimen Screening Creep Equipment

All wiring, helium, and water connections to the six MS stands are complete. Couplings and load bars for these units have been delayed by Applied Test Systems' casting sub-vendor. Deliveries to GE are now estimated for mid-June. Lower load bars are receiving highest priority because they contain the O-rings which seal the lower end of the retort systems. The retorts cannot easily be sealed for preliminary leak checks until these seals are available.

2.3.3 Thermal Stability Equipment

All eight stands, furnaces, and retorts have been received. The vendor, Satec Systems, provided a field service engineer on site

for two days to inspect the delivered retort systems and instruct GE personnel in their proper installation. Four systems are completely installed; one small stainless steel fitting remains to be delivered for the helium supply lines to each of the other four units.

2.3.4 Single Specimen Creep/Rupture Equipment

There have been no further equipment deliveries from ATS during this quarter. Of the 30 stands and 20 retort systems already delivered, 10 have been assembled, installed, and their load trains calibrated. These 10 units will be utilized for screening creep measurements on the TZM alloy as mentioned in previous reports. Of the 10 units set up, three have had their retort systems fully assembled and connected to all utilities; they were operated during check-out of the temperature control system discussed below.

The LVDT's have been connected to one extensometer and tied into the creep monitoring system. This was done to verify proper operation of the channel scanner and data logger.

2.3.5 Temperature Control System

Hardware installation and checkout were completed during March, 1978. Also, the first installation of software and its shake-down were accomplished. The system was used to control three ST furnaces during this checkout and performed as planned. Minor "clean-up" of the software is now underway by Research Incorporated; this does not include any essential features of system operation, but only such details as output formats being revised to a more convenient, less repetitive form. Two laboratory technicians were given intensive training by the software engineer. The temperature control system is currently operational.

2.4 TASK 4 - SIMULATED REACTOR HELIUM ENVIRONMENT FACILITIES AND EQUIPMENT

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

The changes to the purification system design described in the last Quarterly Report, Progress Report for Period October 1 to December 31, 1977 (C00-2975-20), were incorporated into the Pumping, Purification, and Gas Analysis sub-assembly for Loop 1. Construction of this sub-assembly was completed during January; it was shipped to Schenectady and has been installed in the HTRMTL (see the discussion for Task 3).

The revised flow schematic for the gas system is shown in Figure 2.3. This schematic includes the changes in the purification system; it replaces the schematic included as Figure 2.2 of the Quarterly for the period of July 1 to September 30, 1977 (C00-2975-17).

Fabrication of the pumping, purification, and gas analysis sub-assembly plus the instrumentation and control consoles for Loop 2 is about 70% complete. Also fabrication of the helium distribution subassemblies for single specimen creep/rupture stands No.s 35-40 and 40-45 is nearly complete. Construction of all of these sub-assemblies will be finished during the next quarter.

2.4.2 Simulated Reactor Helium Chemistry

During this quarter, the helium chemistry tentatively recommended for the screening tests, as discussed in the Progress Report of the Period January 1 to March 31, 1977 (C00-2975-10) was revised slightly. The tentative helium chemistry, chosen to represent the primary circuit coolant for a direct cycle helium turbine system and/or a nuclear process heat system with an intermediate heat exchanger was basically a compromise between the chemistries chosen for these two systems by Graham, Everett, et al⁽¹⁾. Since the impurity levels for the two different systems derived in Reference 1 were not greatly different, and particularly since the relevant impurity ratios (relevant from a thermodynamic standpoint) were quite similar, the compromise was a reasonable one to make. The modifications that were made to impurity levels derived in Reference 1 are discussed in Quarterly Report C00-2975-10.

FIGURE 2.3 REVISED FLOW SCHEMATIC, CONTROLLED PURITY HELIUM SYSTEM

The preliminary impurity levels selected were (all in micro-atmospheres) 450H_2 , $1.5\text{H}_2\text{O}$, 30CH_4 , 60CO , 0.3CO_2 , $<20\text{N}_2$.

The final impurity levels recommended for the screening phase environment, for tests of both nuclear process heat candidate alloys and direct cycle helium-turbine candidate alloys were developed as described in the following discussion:

First, the hydrogen level was simply rounded off to 400 microatmospheres.

The $\text{H}_2/\text{H}_2\text{O}$ ratio was chosen as 200:1, as a reasonable compromise between the ratio of 200:1 to 400:1 proposed by (1) and the considerably lower ratio of about 20:1 proposed by General Atomic Co.⁽²⁾ Thus, the proposed water level is 2 microatmospheres.

The CO level in an HTGR is set primarily by reaction of the water with graphite. The reaction rate is a function of the primary coolant density, its flow rate, the water level in the primary coolant, the type of graphite and the graphite temperature (and probably other variables). A reasonable value of percent of water reacting with graphite to form CO and H_2 for a large HTGR system is 90-95%. Thus, to retain 2 μatm H_2O in the primary coolant steady state, water ingress must be sufficient to produce 20 to 40 μatm CO (upon reaction of the water with graphite). Since the higher value would lead to the worse carburization and therefore is probably the "worst condition" case, the CO level was chosen as 40 μatm .

The correct CO_2 level is extremely difficult to determine since for most (all) previously operated reactor systems, under most operating conditions, the CO_2 level was below the detection limits for the monitoring equipment used. There is more evidence to suggest that the CO_2 level is close to that predicted by the

(2) R. D. Burnette, General Atomic Co., Unpublished, private communication,

water shift reaction:

Since the equilibrium constant for this reaction in the temperature range of interest (700-1100°C) is nearly unity (i.e. $\Delta G^\circ \approx 0$), the CO_2 level calculated is about 0.2 μatm .

Previous work suggests that the H_2/CH_4 ratio should be between 10:1, for low temperatures ($\sim 700\text{--}800^\circ\text{C}$, Reference (1)) and 100:1 at high temperatures (900-1000°C, see reference (3)). From these ratios, CH_4 levels of 4 to 40 μatm would be predicted. The value of 20 μatm CH_4 was chosen as a reasonable compromise.

The nominal impurity levels to be used in the screening tests are given in Table 2.8 where they are compared to the nominal values recently chosen by Federal Republic of Germany engineers as a representative environment for PNP (Prototypanlage Nuklear Prozesswärme - Prototype Nuclear Process Heat) materials testing and to the nominal values of the High Temperature Materials Program/General Atomic Co. (HTMP/GAC) Phase IV tests (GAC representative DCHT environment). Table 2.9 lists the impurity ratios of importance in terms of the oxidizing/reducing and carburizing/decarburizing reactions listed in Table 2.10.

The similarities between these environments are made even more obvious by a comparison of oxygen potential and carbon activity calculated, for each test gas chemistry, at 727°, 927°, and 1127°C, for several reactions by methods indicated in Table 2.11. The results, as given in Table 2.12, at any given temperature and reaction, differ for the different test gas chemistries, by less than a factor of 3.5. This difference is well within the error limits for prediction of relevant environments, is probably within the scatterband of composition variation during test runs, and in no case do the different environments result in different predicted results (from thermodynamic considerations).

- (3) J. Englehard and K. Kruger, "Investigation of the Impurities and Fission Products in the AVR Coolant Gas at an Average Hot-Gas Temperature of 950°C; Nuclear Eng. & Design. V.34 (1975), No. 11, p.85

TABLE 2.8

HELIUM IMPURITY LEVELS FOR MATERIAL PROPERTIES TESTSCOMPARISON OF PNP, HTMP/GAC PHASE IV, GE

IMPURITY	IMPURITY CONC. (μ ATM)		
	PNP	HTMP/GAC PHASE IV	GE
H ₂	500	500	400
H ₂ O	1.5	1.5	2.0
CO	15	50	40
CO ₂	(EQUIL.)	(EQUIL.)	0.2
CH ₄	20	50	20
N ₂	<5	--	<20

TABLE 2.9

HELIUM IMPURITY RATIOS FOR MATERIALS PROPERTIES TESTSCOMPARISON OF NPH, HTMP/GAC PHASE IV, GE

IMPURITY RATIO	PNP	HTMP/PHASE IV	GE
P_{H_2}/P_{H_2}	333	333	200
P_{CO}/P_{CO_2}	~150	~50	200
P_{CO}^2/P_{CO_2}	0.002	0.003	0.008
$P_{CO}P_{H_2}/P_{H_2O}$	0.005	0.017	0.008
$P_{CH_4}/P_{H_2}^2$	80	200	120
$P_{CO} P_{CH_4}/P_{H_2} P_{H_2O}$	0.4	3.3	1.0

TABLE 2.10

PARTIAL PRESSURE RATIOS RELEVANT FOR OXIDIZING/REDUCING AND
CARBURIZING/DECARBURIZING REACTIONS

PARTIAL PRESSURE RATIO	REACTION
P_{H_2}/P_{H_2O}	$H_2O = H_2 + \frac{1}{2}O_2$
P_{CO}/P_{CO_2}	$CO_2 = CO + \frac{1}{2}O_2$
P_{CO}^2/P_{CO_2}	$2CO = C + CO_2$
$P_{CO}P_{H_2}/P_{H_2O}$	$CO + H_2 = C + H_2O$
$P_{CH_4}/P_{H_2}^2$	$CH_4 = C + 2H_2$
$P_{CO}P_{CH_4}/P_{H_2}P_{H_2O}$	$CO + CH_4 = C + H_2 + H_2O$

TABLE 2.11

EQUATIONS FOR OXYGEN AND CARBON POTENTIALS OF TEST HELIUM ENVIRONMENTS

ACTIVITY (POTENTIAL) CONSIDERED	REACTION	ACTIVITY (POTENTIAL) EXPRESSION
P_{O_2}	$2H_2 + O_2 \rightleftharpoons 2H_2O$	$P_{O_2} = \left(\frac{P_{H_2O}}{P_{H_2}} \right)^2 \text{EXP } (\Delta G^0/RT)$
a_C	$C + 2H_2 \rightleftharpoons CH_4$	$a_C = \frac{P_{CH_4}}{(P_{H_2})^2} \text{EXP } (\Delta G^0/RT)$
a_C	$C + H_2O \rightleftharpoons CO + H_2$	$a_C = \frac{P_{CO}P_{H_2}}{P_{H_2O}} \text{EXP } (\Delta G^0/RT)$
a_C	$2C + H_2 + H_2O \rightleftharpoons CO + CH_4$	$a_C^2 = \frac{P_{CO}P_{CH_4}}{P_{H_2}P_{H_2O}} \text{EXP } (\Delta G^0/RT)$

TABLE 2.12

OXYGEN AND CARBON POTENTIALS OF TEST HELIUM ENVIRONMENTS

ACTIVITY	TEMP. (°C/°F)	ACTIVITY VALUE		
		PNP	CIIR/PHASE IV	GE
P_{O_2} $(2H_2O \rightleftharpoons 2H_2 + O_2)$	727/1341	9.4×10^{-26}	9.4×10^{-26}	26×10^{-26}
	927/1701	1.8×10^{-21}	1.8×10^{-21}	5.0×10^{-21}
	1127/2061	2.1×10^{-18}	2.1×10^{-18}	5.7×10^{-18}
a_c $(CH_4 \rightleftharpoons C + 2H_2)$	727	8.1×10^2	20×10^2	13×10^2
	927	5.0×10^3	12×10^3	7.8×10^3
	1127	1.9×10^4	4.6×10^4	2.9×10^4
a_c $(CO + H_2 \rightleftharpoons C + H_2O)$	727	1.8×10^{-3}	6.2×10^{-3}	2.9×10^{-3}
	927	1.2×10^{-4}	4.2×10^{-4}	2.0×10^{-4}
	1127	1.8×10^{-5}	6.1×10^{-5}	2.9×10^{-5}
a^2_c $(CO + CH_4 \rightleftharpoons 2C + H_2 + H_2O)$	727	1.2	3.6	2.0
	927	0.79	2.3	1.3
	1127	0.58	1.7	0.91

Table 2.12 also indicates the relatively low oxidation potential of these environments (for the CO and CO₂ levels chosen for the General Electric tests, the oxidation potentials from the reaction $\text{CO}_2 \rightleftharpoons \text{CO} + 1/2 \text{O}_2$ are similar to the oxidation potential from the reaction $\text{H}_2\text{O} \rightleftharpoons \text{H}_2 + 1/2 \text{O}_2$). The CH₄ decomposition results in extremely high carbon activities which increase with temperature. The reaction of CO and H₂ gives fairly low carbon activity which decreases with increasing temperature, while the reaction of CO and CH₄ listed last in Table 2.12 results in carbon activities of approximately unity. (A carbon content of unity or greater is a necessary condition for carbon deposition, while a carbon activity of about 0.1-0.001 is required for carbide formation in common high temperature alloys).

Finally, Table 2.13 indicates that even if the CH₄ level is decreased by a factor of 100 (at constant hydrogen levels), methane decomposition is still strongly carburizing (the minimum carbon activity is 20 at 727°C). Water levels, however, are much more critical. A factor of ten decrease in water level (at constant hydrogen levels), which could possibly occur due to water depletion in test rigs or in stagnant or low flow regions in reactor systems or test rigs, or a factor of 10 increase in H₂ level (for constant H₂O), can result in oxygen activities lower than that required for Cr₂O₃ stability, particularly at higher temperatures. (The methods of activity calculations are indicated in Table 2.14.) The presence or absence of stable oxides is expected to be of considerable significance with respect to carburization/decarburization reactions.

2.4.3 Simulated Reactor Helium Instrumentation, Control Systems, Start-up and Commissioning Procedures

2.4.3.1 Instrumentation

The Antek 320 gas chromatograph has been installed and the carrier gas, calibration gas and exponentially diluted gas lines

TABLE 2.13

OXYGEN AND CARBON ACTIVITIES

TEMP. (°C)	$P_{CH_4}/P_{H_2}^2$	a_c	P_{H_2}/P_{H_2O}	P_{O_2} (ATM)	P_{O_2} FOR Cr_2O_3 STABILITY ^(A)
727 (1341°F)	200	2×10^3	330	9×10^{-26}	$> 3.4 \times 10^{-30}$
	2	20	3300	9×10^{-28}	
927 (1701°F)	200	1.2×10^4	330	1.8×10^{-21}	$> 1.0 \times 10^{-23}$
	2	1.2×10^2	3300	1.8×10^{-23}	
1127 (2061°F)	200	4.6×10^4	330	2.1×10^{-18}	$> 5 \times 10^{-19}$
	2	4.6×10^2	3300	2.1×10^{-20}	

(A) FOR $a_{CR} = 0.3$

TABLE 2.14

CALCULATIONS OF OXYGEN & CARBON ACTIVITIES

$$p_{\text{O}_2} = \left(\frac{p_{\text{H}_2\text{O}}}{p_{\text{H}_2}} \right)^2 \text{EXP} \left(\Delta G^0/RT \right)$$

$$p_{\text{O}_2} = a_{\text{O}_2} = \left\{ (a_{\text{CR}})^{-2} \text{EXP} \left(\Delta G^0/RT \right) \right\}^{2/3}$$

$$a_{\text{C}} = \frac{p_{\text{CH}_4}}{p_{\text{H}_2}^2} \text{EXP} \left(\Delta G^0/RT \right)$$

have been connected. The entire calibration manifold for the chromatograph is complete, with some modifications having been made to eliminate dead volumes and expedite flows. The instrument will be checked out and calibrated during the next quarter period. The exponential dilution flash to be used in calibration of the gas chromatograph has been characterized and mounted on a magnetic stirring device.

The dew point meter has been installed in the console. Sensors have been inspected, installed, and are currently awaiting calibration by the techniques outlined below. The oxygen cell has been examined, installed, and calibrated. The 16-port scanning valve has been in use for directing calibrating gases to the instrumentation. The 16-port valve is equipped with a helium purge to prevent air from entering in through leaks which may develop in the valve plate seals as a result of extended operation. To accomplish this, a small amount of pure helium is being pirated from the carrier gas line, with the flow rate and pressure at the 16-port valve being controlled by a needle valve.

2.4.3.2 Calibration of Instrumentation

The calibration system for the gas chromatograph has been completed without any changes in design. Calibration of the oxygen cell was accomplished by routing the calibration gas into the oxygen probes by using the special channel incorporated in the 16-port valve for this purpose. The calibration gas is a mixture of 400 ppm H_2O and 10,000 ppmv H_2 in Helium. The partial pressure of O_2 determined from the O_2 probe agreed exactly with the value calculated for this gas mixture (e.g. for the H_2/H_2O ratio of this gas mixture) at the O_2 probe temperature of $750^{\circ}C$ (equilibrium conditions assumed).

The calibration of the hygrometer probes awaits the construction of a small stainless steel manifold which will allow gas from any of three cylinders to be flowed to both dew point probes, each vented separately through a bubbler so that the flow rate and pressure at either probe can be measured. The three cylinders will contain:

- o A moisture standard of about 1 ppmv H_2O
- o A moisture standard of about 10 ppmv H_2O
- o A cylinder of nominally dry helium (~ 2 ppmv H_2O)

2.4.3.3 Preparation of Purification Components

The materials to be used for the loop gas purification have all been regenerated and/or purified by the processes summarized in Table 2.15. After each process, the purification canister was pressurized to 10 psig with pure helium, sealed, and labelled.

2.4.3.4 System Startup

It is anticipated that adsorbed moisture will be the principal contaminant to be removed after the system is completely assembled and leak-tight. In order to bring the entire system moisture level down to the desired levels (< 1 ppm) as rapidly as practicable, it is planned to evacuate the system as a unit, fill with high-purity helium to 5 psig and then operate in such a fashion that the helium is continuously dried by the purification system and circulated throughout the distribution system. More helium will be fed into the system until discharge pressure stabilizes at approximately 20 psig and return receiver pressure is at 5 psig. At this time, helium will be pumped throughout the distribution system as if it were in normal operation. Flow will be through each retort, or dummy retort, into the return lines and thence to the return receiver in the normal fashion. Also, flow will be established in all sense lines to the 16 port scanning valve and thence through the gas analysis station of the PP&A and to the return receiver. The fill gas manifold and connecting lines will be subjected to continuous flow via the temporary line from the discharge (high pressure) receiver.

Flow, counter to the direction which would occur during normal system operation, will be established in each impurity gas addition line and manifold and in the purge and back-up gas manifolds and their associated gas purification devices.

Table 2.15

PREPARATION OF PURIFICATION SUBSYSTEM

<u>Purification Unit</u>	<u>Vacuum Conditions Time Required to Pump Down to $\leq 10^{-3}$ Torr</u>	<u>First Gas</u>	<u>Temp. (°C)</u>	<u>Flow (ℓ/min)</u>	<u>Time (Hr.)</u>	<u>Second Gas</u>	<u>Temp (°C)</u>	<u>Flow (ℓ/min)</u>	<u>Time (Hr.)</u>
Activated Charcoal	~ 24 Hrs	High Purity Helium	180	0.5	24	--	--	--	--
Copper	~ 30 min	Helium with 500ppm H ₂	250	3.0	4	High Purity Helium	270	3	6
Magnesium Perchlorate	~30 min	High Purity Helium	100	0.5	24	--	--	--	--
Ridox	~30 min	Helium with 5% H ₂	195	2.0	24	High Purity Helium	195	3	4
Copper Oxide	~30 min	High Purity Helium	220	2.0	6	--	--	--	--
Ascarite	~60 min	High Purity Helium	100	0.5	24	--	--	--	--
Molecular Sieve	~20 hr. at 180°C	High Purity Helium	100	0.2	24	--	--	--	--
Oxalic Acid	---	Helium with 1000ppm H ₂ O	25	0.5	4	--	--	--	--

With this flow pattern established throughout the entire system, the moisture, O_2 , and N_2 removal portions of the purification package of the PP&A will be activated to simultaneously remove desorbed moisture and residual air. Progress of both these processes will be measured with built-in instrumentation. When acceptably low levels have been attained, circulation will be stopped and the system will be left with a charge of pure, low-oxygen, low-moisture He, ready for the commissioning experiments detailed below.

2.4.2.5 Commissioning Procedure

The gas chemistry experiments to be conducted during the commissioning phase of the project have been finalized and outlined in detail. Two sets of experiments are to be conducted to characterize gas chemistry phenomena in the loop and retorts respectively. These measurements will be carried out along parallel schedules.

The following experiments will be made on the loops, after the system has been satisfactorily evacuated, filled with He and purified as described below:

1. Oxygen Inleakage Measurements - The amount of atmospheric O_2 leaking into the loop can best be measured before the addition of reducing gases such as H_2 and CO, and is thus incorporated into the commissioning procedure. This measurement will be made by circulating the loop He through the purification system to lower O_2 and H_2O concentrations to a stable level. The shape of the concentration vs. time curves should indicate when most of the desorption has occurred. The purification components will be closed off, and with the gas circulating through the loop, the measurement of O_2 concentration vs. time will be made. The slope of this line should indicate the O_2 inleakage rate, and should remain independent of flow rate.

2. Sense Line Purge Time Determination - All sense lines are routed from the 16-port valve to the analysis instrumentation through a common line and manifold. Since this system contains several dead volumes (e.g. in pressure gage tubes) the amount of time required to completely purge out the lines after selecting a new channel cannot be calculated directly by exponential volumetric dilution techniques. An empirical value is probably the most reliable. To establish this value, a gas stream containing sizeable concentrations of two impurities, A and B, will be passed through the analysis system and analyzed. The next stream admitted to the analysis system will contain only impurity A, and this gas will be purged through for a given time, then analyzed for residual B content. This experiment will be repeated until a satisfactorily low amount of B is detected.
3. Gas Addition Parameters - The rates at which deliberate impurity gas additions alter the overall loop gas composition must be accurately known in advance. This will be determined by adding these gases to the loop in both the high and low flow rate modes, and observing the resulting concentration changes. The setting of the micrometer valve will be changed, and the experiment repeated. These data will be converted into a plot of flow rate of impurity gas vs. micrometer setting. The mixing rates for these gases will also be established by observing the time required to reach the new equilibrium value after each addition.
4. Impurity Gas Purification Parameters - The rates at which impurity gases are removed by the purification system will be evaluated by passing loop gas over various getters at a known flow rate, and observing the concentration decrease. The efficiency of each getter

will be calculated, and the effects of side reactions from the purification processes will be observed to determine how the overall loop chemistry will be affected.

During the time that the above four experiments are being run the following tests will be performed on the retorts.

5. Impurity Desorption Measurements During Bakeout - This test is designed to determine which impurities desorb from the retort and coupling surfaces and at what levels during the time the temperature is raised from ambient to the test temperature and purge gas is flowing through the retort. The levels of O_2 , H_2O and hydrocarbons which will desorb, and which could react with the test specimens, will be closely monitored during this test by using the sense lines and console analysis equipment.
6. Programmed Temperature-Time Bakeout Experiment - Should the levels of impurities exceed a safe limit during purging at temperature where undesirable reactions could occur during a normal linearly ramped temperature program, the use of a modified temperature-time schedule may be necessary. This program will be drawn up based on the experimental findings of the bakeout experiment, and will seek to either slowly release desorbed species to keep them at low levels throughout the bakeout, or to rid them rapidly, producing high concentrations but at temperatures where reactions with the test specimens will not occur significantly.
7. Oxygen Inleakage Measurements - A baked out retort containing pull rods and possibly a dummy specimen will be swept with pure He containing no reducing gases. The O_2 content of this gas will be measured. The retort

will then be isolated with the gas pressurized to that level to be used during normal operation. The O_2 level increase will be measured after several days, and translated into an inleakage rate.

8. Conditioning a Retort - A metal retort and couplings will be conditioned with a mixture of 400 ppmv H_2O and 10,000 ppmv H_2 in He at the temperature at which the retort is to be used. This process is designed to rapidly preoxidize any metal surfaces which would be oxidized at a much slower rate if exposed directly to the test gas. A conditioned retort should deplete the test gas of H_2O , CO_2 , CH_4 etc. much less than an unconditioned retort.
9. Determination of Conditioning Effects - It will be necessary to insure that a conditioned retort does not cause serious changes in the impurity levels of the test gas. This will be accomplished by passing a mixed gas composition through a heated retort containing couplings, and observing any changes in gas composition. The measurements will be compared to those on an unconditioned retort. These results will determine to what extent, if any, adverse effects can result from conditioning.

The final phase of the commissioning procedure will be the attainment of the desired impurity levels in the loop. To insure that this chemistry can be maintained under stable circumstances, the loop will be run for a week or two to note how much effort is required to stay within the required impurity concentration limits. During this trial period, mock disturbances corresponding to such events as a retort addition, retort removal, stuck valve, opening of the wrong getter, etc. will be staged to determine our capabilities in handling them.

2.5 TASK 5 - MECHANICAL PROPERTY EVALUATION

2.5.1 Test Specimen Machining

This activity for both DCHT and NPH alloys was initiated at the chosen vendor's shop during this quarter. More detailed discussion is included under Task 2.

2.5.2 Strain Determination for Multiple Specimen Creep Specimens

A device for measuring lengths between gauge marks of screening creep specimens has been designed and is shown schematically in Figure 2.4. Key elements in this device are the precision translating tables. These units, manufactured by Schneeberger (Switzerland), provide zero-play, elastically pre-loaded roller bearings for precise, low-friction motion. The sample to be measured will be firmly attached to the longer table, and the pseudo-knife edge mounted on the smaller table will be engaged in one of the reference grooves machined into the specimen shoulders. At this time, the electronics package reset circuit will be activated, and the Light Emitting Diode display will indicate all reference marks (zero readings). The knife-edge table will then be retracted and the specimen table translated until the second reference groove is engaged. As this translation occurs, the glass scale attached to the specimen table passes through a stationary encoder (reading head). The encoder, scale, and electronics are manufactured by Teledyne-Gurley of Troy, NY, and are capable of detecting translation of the specimen table to the nearest 1.27 microns (50 microinches). The electronic output of the unit is in binary coded digital (BCD) form and will ultimately be interfaced with a computer system for semi-automatic data acquisition and storage. During the course of the screening program, this measurement will be repeated on each specimen at 1,000-hour intervals. At the end of the test, and periodically during the tests, stored data will be processed into creep strain versus time and reported in graphic and tabular form.

FIGURE 2.4 SCREENING CREEP ELONGATION MEASURING DEVICE (SCHEMATIC)

2.6 TASK 9 - DATA MANAGEMENT AND PROGRAM MANAGEMENT

2.6.1 Data Management - (R. B. Hand)

2.6.1 Data Acquisition Procedures

o Data Encoding Coordination

A minor error in the encoding logic was found. This has been corrected to permit the unambiguous identification of previously exposed specimens and specimens examined after testing. Also, changes have been made to the encoding forms: 1) provide for encoding compound concentrations, e.g. Y_2O_3 ; combined analyses, e.g. Ta + Nb; indicate if concentration is less than or greater than a given value; 2) provide for encoding gas flow rates during testing; 3) provide an automatic system for calculating and presenting averages and standard deviations for gas chemistries, flow rates and temperatures during a test; 4) provide improved material identification and traceability.

Encoding forms and instructions for corrosion/erosion data have been developed. It was decided to combine the Metallography/Microstructure data set with the Test Results data set.

o Encoding Instructions

The changes and additions indicated above have made extensive editing of the encoding forms and instructions necessary.

2.6.1.2 Data Storage and Retrieval Procedures

The changes and modifications indicated above have required a major modification of the file structure of the data management system, PAMMED. Determination of test durations and machine usage has been improved.

o Data Storage Procedures

Modification of the data storage computer program is required to incorporate the indicated changes. This work is progressing. All data will be stored on disks to facilitate retrieval. Also, all data will be archived to prevent loss. The archive system provides three backup tapes to achieve even greater insurance against loss.

The program to initialize the file structure is complete.

The program for maintaining the data base has the following features complete:

- 1) Add new data
- 2) Edit existing data
- 3) Delete existing data
- 4) Generate time differences for the Purchasing data set

Work in progress on this program consists of:

- 1) Calculation and presentation of test duration and machine usage.
- 2) Statistical analysis and presentation of averages and standard deviations of gas chemistries, temperatures and flow rates during tests.

o Data Retrieval Procedures

As soon as the modified material description forms are complete, simple selective retrievals based on material identity numbers (MINO) and data sets can be made; i.e., all data in the data bank may be retrieved, or all data for a given material identity number (MINO) or parts thereof as defined by a data set may be retrieved.

The next major step in retrieval procedures will make possible complex selective retrievals based on any set of criteria; e.g., alloy, material form, test property, etc. This step will require a major effort and time expenditure.

2.6.2 Program Management

The second major program review by the Advanced Gas Cooled Nuclear Reactor Materials Program Advisory Review Board was held on March 7 and 8, 1978. The Review Board members are: L. P. Jahnke (Consultant), Chairman, Dr. L. F. Coffin (General Electric Corporate Research and Development), Dr. L. W. Graham (High Temperature Materials Program), Professor P. Kofstad (Professor, Department of Chemistry, University of Oslo), Professor H. Nickel (Head, Institute für Reaktor Werkstoffe, Kernforschungsanlage, Jülich, FRG), C. T. Sims (General Electric Gas Turbine Division), and G. M. Slaughter (ORNL).

Presentations of progress over the past year and plans for the present year, for the Advanced Gas Cooled Nuclear Reactor Materials Program were made by the members of the program team, which included C. L. Ehde, Dr. D. W. McKee, Dr. R. B. Hand, E. W. Ross, Dr. D. H. Baldwin, R. K. Elton, Dr. R. M. Goldhoff, S. Z. Hayden, Dr. O. F. Kimball and R. G. Frank. Other program members present were Dr. R. V. Hillery, Dr. A. W. Urquhart. R. Stepien led a tour of the HTRMTL facility for the Advisory Review Board.

The presentations were favorably received by the Advisory Review Board. Overall all, the review board commented favorably on the progress made and on the program plans. Recommendations that were made by the Review Board will be formally published in a separate report.

Other meetings which were held during the quarter included an internal Program Appraisal and Review (PAR) meeting with General Electric management (Jan. 9, 1978) and technical exchange meetings with the Institute für Reaktor Werkstoffe, Kernforschungsanlage, Jülich, FRG (Feb. 17-21, 1978); Brown, Boveri, CIE, Mannheim, FRG (Feb. 22, 1978); Hochtemperatur-Reaktorbau (Feb. 22, 1978); and High Temperature Materials Programme Wimborne Dorsett, England (Feb. 23, 24, 1978).

A meeting was held with the DOE Program Manager at DOE, Germantown, Maryland on April 5, 1968. The purpose of the meeting was to present to DOE the schedular impact on the program as a consequence of the current FY1978 funding limitations. At the present level of funding for FY1978, initiation of the screening test program (Tasks 5, 6, 7) cannot begin before FY1979 funding becomes available. A revision (No. 3) to the Master Program Schedule was prepared to reflect this change and is presented in Figure 1. Other changes in the schedule that were made are as follows:

<u>TASK</u>	<u>TITLE</u>	<u>CHANGE</u>	<u>REASON</u>
2	Materials Procurement and Characterization	Two month delay in procurement of 2 NPH plant alloys from April, 1978 to June, 1978; will also result in delay of characterization and joining sub-tasks 2.2, 2.3 to July	Strike at Vendor's
3	Test and Analysis Facilities and Equipment		
4	Simulated Reactor He Facilities and Equipment		
	Loop 1	Completion of commissioning extended to 8/1/78	Increased time has been required to leak check system
	Loop 2	Completion of commissioning extended to 6/1/79	Intentional delay because of FY1978 funding limitations. No impact on program
9	Data Management and Program Management	Completion of final Data Management procedures delayed to December, 1978	Intentional delay because of FY1978 funding limitations. No impact on program.
		Completion dates for Topical Reports 2, 3, 4	Dates changed to reflect change in Testing Schedule

3.0 WORK TO BE COMPLETED DURING THE NEXT QUARTER

- o The remaining two nuclear process heat alloys will be received.
- o Test specimen machining for characterization tests will be completed.
- o Tensile and rupture tests for DCHT materials will be performed to check for conformance with specification requirements.
- o 300 hour rupture tests for DCHT alloys, part of the characterization tests for these alloys, will be completed.
- o Leak checking and leak repairing for "helium loop" number 1 will be completed.
- o Initial loop pump down, helium charging, and shakedown will be finished.
- o The remaining subassemblies for loop number 2 will be finished.
- o Data acquisition procedures and data storage/retrieval procedures will be defined.