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ADVANCED GAS COOLED NUCLEAR REACTOR MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

PROGRESS REPORT
FOR PERIOD

APRIL 1, 1979 - JUNE 30, 1979

BY
PROJECT STAFF

GENERAL ELECTRIC COMPANY
ENERGY SYSTEMS PROGRAMS DEPARTMENT
SCHENECTADY, NEW YORK 12345

GENERAL ELECTRIC

JANUARY 25, 1980

Prepared for

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

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FOREWORD

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, under Contract EY-76-C-02-2975. 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 Principle Investigator, with responsibility for overall technical coordination of the Program, is Dr. O. F. Kimball, Manager, Gas Reactor Materials, assisted by Dr. J. F. Stubbins, Materials Engineer, Gas Reactor Materials. Other major contributors to the Program are:

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Turbine (DCHT) - (Mr. E. Ross)
Program Data Analysis - (Dr. R. B. Hand)
- Dr. S. Yukawa, Large Steam Turbine-Generator Operation,
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Systems (NPH) - (Mr. S. Z. Hayden and
Mr. R. A. Ellis)
- Mr. C. L. Ehde, Advanced Energy Programs, Cincinnati, Ohio
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Project Manager - Materials Testing in Simulated Reactor
Helium - (Dr. D. H. Baldwin, Mr. R. K. 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, 1979, through June 30, 1979, 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) Nuclear Process Heat (NPH) and Direct Cycle Helium Turbine (DCHT) applications, in terms of the effect 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 status of the simulated reactor helium supply system, testing equipment, and gas chemistry analysis instrumentation and equipment. The status of the data management system is presented. In addition, the progress in the screening test program is described.

OTHER REPORTS IN THIS SERIES

QUARTERLY PROGRESS REPORTS

September 23, 1976 to December 31, 1976	C00-2975-3
January 1, 1977 to March 31, 1977	C00-2975-10
April 1, 1977 to June 30, 1977	C00-2975-13
July 1, 1977 to September 30, 1977	C00-2975-17
October 1, 1977 to December 31, 1977	C00-2975-20
January 1, 1978 to March 31, 1978	C00-2975-21
April 1, 1978 to June 30, 1978	C00-2975-22
July 1, 1978 to September 30, 1978	C00-2975-25
October 1, 1978 to December 31, 1978	C00-2975-28
January 1, 1979 to March 31, 1979	C00-2975-31

TOPICAL REPORTS

1. Selection of Candidate Alloys	C00-2975-16
Volume 1 - Advanced Gas Cooled Reactor Systems Definition by M. Marvin	October 31, 1978
Volume 2 - Selection of Alloys for Screen- ing Creep and Structural Stability Studies by R.M. Goldhoff, R.V. Hillery, and S. Yukawa	December 31, 1978
Volume 3 Selection of Surface Coating/ Substrate Systems for Creep and Structural Stability Studies by D.A. Grey	To Be Released

Section 1.0

INTRODUCTION

ADVANCED GAS COOLED NUCLEAR REACTOR
MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

1.0 INTRODUCTION

The Very High Temperature Reactor (VHTR) Nuclear Process Heat (NPH) and Direct Cycle Helium Turbine (DCHT) Systems are being developed to provide an expanded energy capability and to facilitate conservation of or substitution for scarce energy resources. 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 NPH and DCHT applications, the need is evident for materials testing in the proper helium environment in the temperature range from 750° - 1050°C (1382° - 1922°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 and thermal exposure on:
 - Surface stability ("corrosion" resistance) in 10,000 hours.
 - Structural stability in 10,000 hours - changes in microstructure and tensile, impact and fatigue properties.
 - Creep - rupture properties in 15,000 hours.
 - 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 NPH and 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 covering the period September 23, 1976 - December 31, 1976, report number 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 topical report number C00-2975-16, Volume 2, dated December 31, 1978 and entitled: Selection of Alloys for Screening Creep and Structural Stability Studies.

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 procurement of all DCHT materials for the screening test program was completed by June 1978.

2.2.1.2 Materials Characterization

This subtask was completed and the final results reported in the Progress Report for July 1, 1978 - September 30, 1978, report number C00-2975-25.

2.2.2 Nuclear Process Heat (NPH) Alloys - (S.Z. Hayden and R.A. Ellis)

2.2.2.1 Materials Procurement

The procurement of all NPH materials was completed by September 1978.

2.2.2.2 Materials Characterization

The materials acceptance and characterization testing was completed in the quarter ending in December 1978, and the final results reported in the Progress Report for January 1, 1979 to March 31, 1979, report number C00-2975-31.

2.2.2.3 Materials Joining Studies

The final results for Vareststraint tests were covered in the quarterly report for October 1, 1978 to December 31, 1978, report number C00-2975-28.

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

2.3.1 Multi-Specimen Creep (MSC) Equipment

Revised furnace windings designed to shorten the hot zone length from 99.1 to 99.5 cm (39 to 36 inches) were received from the vendor, Applied Test Systems (ATS). This version was necessitated by the unacceptable thermal gradient in the ceramic MSC retorts with the longer hot zone length. The revised furnace windings have been installed on one stand for testing.

One of the MSC stands, with an Inconel 601 retort for the testing at 750°C (1392°F), has undergone load train calibration. This calibration is done simultaneously on all six load trains at loads approximating their service load. This method was adopted due to the possibility of interaction between the load trains under load.

The device for measuring length changes in multi-specimen creep specimens was calibrated against a Pratt and Whitney super micrometer and found to be accurate within ± 0.00127 mm (± 0.00005 inch) over a 6.35 cm (2.5 inch) range. The device readout is in inches, and for this reason, inches are the primary unit and the metric numbers are from conversion.

Final mechanical revisions to the specimen holder of this unit have been made and measurements begun on screening creep test specimens. In order to determine the accuracy and repeatability of measurements, seven randomly selected specimens were independently measured five times each by five different operators. Two dimensions were measured on each specimen; the distance between the shoulder reference grooves at positions diametrically opposed around the sample, using the machined slot in the

specimen ends as the rotation reference. The sample was physically removed from the device between measurements. Instrument and specimen temperatures were within 23.3-23.9°C (74-75°F) at all times.

The repeatability of measurements was excellent, with all five measured values falling within the vendor's claimed accuracy limit of ± 0.00127 mm (± 0.00005 in) in more than 97% of the fourteen sets of readings obtained. In cases where measurements exhibited a range of more than the accuracy limit of the instrument, cleaning and remeasurement of the specimens always reduced that range to the accuracy limit or less.

Accuracy of measurements was determined by comparing the values of the same dimension as measured by different operators. First, the five values reported by a given operator were averaged. Then, these average values were averaged and their standard deviation determined. The standard deviations also were expressed as per cents of their respective means to normalize the results which are given in Table 2.1. It will be noted that standard deviations of 0.00010 inches or more were obtained for each of the three smaller samples, while all of the four larger specimens exhibited lower standard deviations. When normalized for length, these results indicate that the expected standard deviation for small specimens (DCHT alloys) is 0.00033 in./in. strain and for large specimens (NPH alloys) the corresponding value is 0.00017 in./in.

2.3.2 Thermal Stability Test Equipment

With the replacement of a burned-out winding in the Aging Corrosion (AC)-8 furnace, all eight thermal stability stands have been rendered operational. AC-2 and AC-1 were loaded with the 1,000- and 3,000-hour and 6,000- and 10,000-hour samples, respectively, for exposure at 950°C. The corresponding furnace loads for 1050°C have been installed in AC-8 and AC-7. The other four AC stands are on test with specimens slated for 1000-, 3000-, 6000- and 10,000- hour exposures at 750°C. (1382°F) and 850°C (1562°F).

2.3.3 Single Specimen Creep-Rupture Equipment

The final three (3) TZM specimens have been installed in the single specimen creep-rupture (ST) stands, ready for loading and tie-in with the loop. These are the last three (3) of nine (9) TZM screening creep tests which are being carried out

TABLE 2.1

GROOVE-TO-GROOVE MEASUREMENTS OF SEVEN
RANDOMLY SELECTED SCREENING CREEP SPECIMENS

Specimen Size	Orientation	Mean (inches)	Standard Deviation (inches)	Standard Deviation (% of mean)
B ^(a)	0°	1.75510	0.000116	0.0066
	180°	1.75516	0.000064	0.0036
B	0°	1.75094	0.000186	0.0106
	180°	1.75115	0.000070	0.0040
B	0°	1.75964	0.000042	0.0024
	180°	1.75970	0.000100	0.0057
C ^(b)	0°	2.35037	0.000049	0.0021
	180°	2.35040	0.000050	0.0021
C	0°	2.36802	0.000026	0.0011
	180°	2.36816	0.000044	0.0019
C	0°	2.36859	0.000048	0.0020
	180°	2.36832	0.000045	0.0019
C	0°	2.36490	0.000069	0.0029
	180°	2.36520	0.000073	0.0031

(a) B-size: 36 mm (1.42 in.) gauge length

(b) C-size: 50 mm (1.96 in.) gauge length

in the ST equipment, due to the alloy's inordinately high strength. (Testing in the MSC equipment alone could not have produced significant stress loads for TZM).

Ten additional ST test stand retort systems have been assembled and installed in preparation for the second or intensive screening phase of the current program.

2.4 TASK 4 - SIMULATED REACTOR HELIUM ENVIRONMENT FACILITIES AND EQUIPMENT (D.H. Baldwin and R.K. Elton)

2.4.1 Loop No. 1 Operation

During April, more than twenty-three (23) days of system operation within chemistry limits were logged. The gas chemistry for this period is listed in Table 2.2.

A voltage excursion in the external power supply network on April 24 resulted in loss of loop He pressure, temperature control system shutdown, and loss of some system cooling water. All tests were switched to back-up gas (He with ~ 500 ppm H_2 and < 3.0 ppm H_2O) and cooled to room temperature. During the loss of loop He pressure, backstreaming air caused the O_2 level in the loop to rise to the point where it could be detected on the gas chromatograph (GC). The threshold for O_2 detection by the GC is ~ 0.2 μatm O_2 , the normal equilibrium O_2 level in the loop is $\sim 10^{-16}$ μatm O_2 . At the time of the incident, ten (10) test stands were on test. These test stands are listed in Table 2.3 with the test hours at the time of the incident. Test specimens in two of the ten retorts were examined during the system outage, but no detectable sign of specimen damage due to the momentarily high loop O_2 level was noted. The Fe and Cr gas chemistry check coupons that are contained in the retorts were removed from one A/C retort and sectioned for metallographical examination. Analysis showed that no oxide scale had formed on the Fe coupon, while there was a detectable scale on the Cr coupon. This finding is consistent with the desired loop chemistry oxidation potential, and indicates that the brief O_2 excursion had little, if any, effect on the specimens on test. If the oxidation potential had been significant, an oxide scale should have formed on the Fe coupon.

The reason for the loss of loop pressure during the power excursion was traced to a solenoid valve in the main system He dump train. It is surmised that the voltage surge was able to jump the switch gap and open the valve allowing system He to escape. The loss of temperature control can be explained by the susceptibility of the micro-computer system to voltage spikes or momentary power outages. In such cases, the memory is perturbed, causing the controlling feature to malfunction.

TABLE 2.2

LOOP NO. 1 GAS CHEMISTRY^(a)

Constituent	Nominal μ atm	Mean μ atm	Std. Dev., μ atm	Std. Dev., % of Mean
H ₂	400	408	31	7.7
H ₂ O	2	2.01	0.20	10.0
CO	40	40.8	4.6	11.3
CO ₂	0.2	0.23	0.04	18.2
CH ₄	20	18.0	2.5	13.8
N ₂	6	4.89	2.4	49.9

(a) April 01 to 23, 1979

The loss of cooling water also was probably due to solenoid valves tripped by the power excursion.

The loop and all tests listed in Table 2.3 were back on line and running again by the end of June, 1979.

2.4.2 Pumping Purification and Analysis Station (PP&A) No. 1

Due to the loop shutdown, two immediate modifications were made to PP&A No. 1, making it less susceptible to further power supply problems:

- (1) Additional isolation transformers were installed in the power supply lines to the temperature control system.
- (2) The He system's main dump valve was replaced due to its erratic performance in post shutdown checkouts.

In addition, the down time was used to make several other modifications to further improve the system's performance. These include:

- (1) Relocation or revision of pressure gauge sense lines to all analytical instruments. Formerly these taps were immediately upstream of the sensing device and provided significant dead space which may account for slow instrument response to sense line changes. All such pressure gauge lines have been either drastically reduced in length or moved downstream of the instrument in question.
- (2) Incorporation of provisions for removing each hygrometer probe from the system in its own housing, pressurized with dry helium or evacuated, for transfer to the planned RCH cart (RCH in this case stands for Remote Calibration of Hygrometers) and vice versa after calibration.
- (3) Addition of a flowmeter, pressure gauge, and back-pressure regulator to the flushing line passing through the impurity addition micro-metering valves. In addition, a portion of this flow can be fed directly to the gas analysis section of PP&A No. 1 via the previously unused analytical Channel 16. This will allow direct measurement of the impurity addition rate through each valve and also help establish new valve settings when control is switched from one bank of valves to the other.
- (4) Addition of a small vacuum pump and valve to the GC sample sub-loop. This will greatly reduce the purge time required when the GC is used to analyze gasses other than loop samples. This had become a problem under the current policy of 100% analyses of new bottled gases received in (HTRMTL) laboratory.
- (5) Addition of check valves in the back-up gas purification station. These valves serve to help isolate the back-up gas manifold from the main helium supply line. Under the revised back-up gas operating scheme, the normal back-up supply pressure is 15 psig, versus 25 psig in the main helium line. This pressure difference causes significant seat leakage in solenoid valve SV-57, allowing system helium to gradually back-fill the back-up manifold to 25 psig. Eventually, this situation would have meant the fill and purge systems would be using loop helium, as well.

TABLE 2,3

TEST CONDITIONS AND STATUS OF LOOP NO. 1
SHUTDOWN, APRIL 24, 1979

Test Stand		Hours on Test	Furnace Load or Test Conditions		
ST-1	Creep	525	TZM	10,000 hr.	950°C
ST-2		377	"	10,000 hr.	1050°C
ST-5		669	"	3,000 hr.	850°C
ST-6		256	"	6,000 hr.	950°C
ST-9		620	"	6,000 hr.	850°C
ST-10		477	"	10,000 hr.	850°C
AC-3	Aging/Corrosion	207	6,000 & 10,000 hr. samples, 850°C		
AC-4		325	1,000 & 3,000 hr. samples, "		
AC-5		485	6,000 & 10,000 hr. samples, 750°C		
AC-6		551	1,000 & 3,000 hr. samples, "		

Also during this time, the following getters in the purification train were regenerated:

Copper Oxide (CuO)
Magnesium Perchlorate No. 2 ($\text{Mg}(\text{ClO}_4)_2$)
Molecular Sieves Nos. 1 and 2
LN₂ cooled activated charcoal trap (ACT)
Oxalic Acid

Along with this, the performance of four (4) of the purification components were evaluated. The results of this check are shown in Table 2.4. The liquid N₂ trap was chosen for testing because it had been difficult to predict an operating lifetime for it due to the difficulty in estimating the surface area. The CuO getter was due for regeneration, and it was thought wise to test how much capacity it had left. The Ascarite #1 and $\text{Mg}(\text{ClO}_4)_2$ #2 canisters are exposed to the highest levels of CO₂ and H₂O respectively (about 15 μatm CO₂ and 140 μatm H₂O), and were therefore also checked. As the table shows, these reagents have not degenerated significantly over their expected lifetime. Based on this information, the regeneration interval for the $\text{Mg}(\text{ClO}_4)_2$ will be extended to every 5 months instead of every 3 months. It is expected that the efficiency of the CuO canister could be improved by operating at a higher temperature (BTS CuO can be used at temperatures up to 475°C (887°F)). During the next period of loop operation, the CuO will be held at 200°C (392°F) instead of 150°C (302°F), and the efficiency at this temperature determined.

2.4.3 Pumping, Purification And Analysis Station (PP&A) No. 2

Due to the high priority assigned to instrumentation calibration and retort testing following the April 24 shutdown of Loop No. 1, there was little construction progress on Loop No. 2 during the report period. Electrical installation of single-specimen creep stand furnaces was nearly completed and fabrication of stainless steel helium lines to join retorts with utility frame sub-assemblies was completed.

2.4.4 Instrumentation

The down time associated with Loop No. 1 shutdown was used to re-characterize and calibrate the hygrometers and the oxygen analyzer. The receipt of an EG&G dew point hygrometer, Model No. 440-C1 with 440-S3 sensor, greatly facilitated this operation. The Shaw dew point hygrometers were calibrated with respect to the EG&G system. Then, as loop chemistry was being re-established prior to test

TABLE 2.4

EFFICIENCIES OF SELECTED PURIFICATION COMPONENTS

Canister	Species Removed	Efficiencies (%) Impurity Removed)	
		Jan. 1979	May 1979
CuG	H ₂	27	31
CuO	CO	69	34
LN ₂ - ACT*	H ₂	89	6
"	CO	80	51
"	CH ₄	80	54
"	CO ₂	65	~100
"	N ₂	--	50
Mg(ClO ₄) ₂ #2	H ₂ O	Approximately 100%. H ₂ O level in main loop held at 1.5 μ atm during removal of 600 μ atm H ₂ .	
Ascarite #1	CO ₂	Approx. 100%. CO ₂ level in main loop held below 0.2 μ atm during removal of 60 μ atm CO.	

*Activated Charcoal Trap

restart, the impurity gas constituents were varied in a controlled manner so that their effect on the oxygen potential could be monitored.

2.4.4.1 Hygrometer Calibration

The two Shaw hygrometer probes in the helium system have been calibrated against the EG&G dew point hygrometer. This instrument has been installed immediately downstream of the Shaw probe which continuously monitors loop gas moisture. The second Shaw probe was simultaneously exposed to the same composition gas and the system allowed to attain dynamic equilibrium. Table 2.5 gives the data obtained; calibration curves were developed from the data. Detailed plots of the data in the region of moisture levels normally encountered in loop operations show that the accuracy of each probe is $\pm 0.2 \mu\text{atm}$ at the nominal desired loop composition of $2.0 \mu\text{atm}$. It was decided to leave the EG&G in the loop to monitor the Shaw probes for possible calibration drift over the next few weeks. This effort has been temporarily delayed, however, by the failure of a power transistor in the EG&G electronics package.

An anomaly has been noted in the behavior of the Shaw probe A-1 located in the gas analysis section. This concerns the pressure sensitivity of the probe readings. During the major portion of the calibration work reported above, this probe and the EG&G were operated at 15 psig in order to decrease the response time of the latter instrument by increasing the concentration of moisture in the sample stream. During normal loop operations, this probe is operated at 5 psig in order to maintain the necessary pressures throughout the entire helium system. Thus, a corrected calibration curve was developed for Probe A-1 by multiplying the calibration curve obtained at 15 psig by the appropriate ratio ($20 \text{ psia}/30 \text{ psia}=0.67$), to compensate for the pressure change to normal conditions. However, when this change was actually made, just prior to resumption of testing, it was observed that the Probe A-1 readings remained on the 15 psig calibration curve instead of dropping to the expected 5 psig values. This behavior is unexplained and contrary to the vendor's claims. This anomaly will be monitored as testing proceeds. By comparison with the other Shaw hygrometer readings, and more importantly with the EG&G dew point data, it should be possible to establish a cause of this phenomenon.

TABLE 2.5

SHAW HYGROMETER READINGS VS. MOISTURE LEVELS MEASURED BY EG&G DEW POINT
HYGROMETER AND CORRECTED TO 2 ATM. ABS. PRESSURE

<u>Date</u>	H ₂ O Determined by EG&G (μ atm at 2 atm. abs.)	<u>Shaw Probe Readings</u>	
		<u>Channel 1</u> <u>Probe D</u>	<u>Channel 2</u> <u>Probe A-1</u>
6/15/79	1.60	210	190
6/14/79	1.65	211	195
6/14/79	1.70	221	200
6/28/79	2.05	243	235
6/18/79	2.68	282	262
6/16/79	2.80	293	262
6/17/79	4.10	328	300
6/16/79	6.30	376	341
6/17/79	9.10	442	387
6/21/79	14.4	556	502
6/21/79	65.0	832	766
6/20/79	180.0	1072	995

2.4.4.2 Oxygen Analyzer Calibration

The reattainment of loop chemistry prior to restart of testing provided an opportunity to establish the performance of the O_2 analysis systems. Due to the multiplicity of reactions which could occur amongst the various constituents of the controlled purity He, the lack of experimental data on reaction kinetics, and the knowledge that each cell has an apparent inleakage of air which would be expected to affect cell output, it was decided that the safest approach was to determine specific cell outputs under loop operating conditions with He of known impurity concentrations passing through the cell. The approach used was to first determine the cell response to H_2/H_2O , in the absence of other impurities, since the ratio of these two was anticipated to be the principal factor influencing cell output. In order to minimize time expenditure, it was decided to obtain data for only the nominal loop levels ($H_2/H_2O = 200/2$) and the extreme values which would be encountered if the loop is controlled within maximum allowable limits. For the H_2/H_2O ratio, these extremes are $H_2(\text{max})/H_2O(\text{min}) = 500/1$ and $H_2(\text{min})/H_2O(\text{max}) = 300/4$.

The next step was to determine the effects of CO/CO_2 in a similar manner by adding appropriate amounts of these impurities to loop gas already containing the nominal levels of H_2 and H_2O . Finally, with H_2 , H_2O , CO , and CO_2 all at their nominal values, CH_4 was added. At each loop composition, oxygen cell readings were obtained at sample flow rates of 50, 240, and 425 cc/min.

The results of this O_2 cell calibration are shown in Table 2.6. Moisture levels given are the averages of the values indicated by the Shaw probes at each step. Note that, as expected, H_2/H_2O is the principal factor in determining cell output, with CO/CO_2 and CH_4 having a much smaller effect. The tendency for a higher sample flow rate to mask the effect of air inleakage is also shown at each step.

The data from Steps 1 and 2 may be compared directly with the manufacturer's predictions for the O_2 cell. Cell output in mV, based on theoretical considerations (i.e equilibrium thermodynamics of the cell at 750°C), is given by:

$$E(\text{mV}) = 955 + 101.5 \log (H_2/H_2O)$$

TABLE 2.6

EFFECTS OF SAMPLE FLOW RATE AND
GAS COMPOSITION ON OXYGEN CELL OUTPUT

Step	He Flow (cc/min)	H ₂	H ₂ O	CO (μ atm)	CO ₂	CH ₄	O ₂ Cell Output (mV)
1	240	113	2.08				1094
2	50	935	1.82				1163
	240	950	1.88				1185
	425	940	1.78				1188
3	50	380	2.23				1131
	240	390	2.22				1148
	425	380	2.24				1151
4	50	388	1.79	41	.04		1138
	240	390	1.79	41	.05		1153
	425	390	1.79	41	.04		1155
5	50	374	1.80	36	.11		1133
	240	370	1.80	36	.11		1149
	425	382	1.80	36	.11		1153
6	50	370	1.81	38	.20		1133
	240	375	1.78	37	.21		1149
	425	370	1.81	38	.21		1149
7	50	370	1.83	38	.21	10	1142
	240	370	1.83	38	.21	10	1154
	425	370	1.82	38	.21	10	1156
8	50	370	1.78	41	.19	24	1148
	240	370	1.79	38	.20	23	1156
	425	370	1.81	38	.20	23	1159

For the 240 cc/min sample flow rate data in Table 2.6, it is possible to substitute in this expression to obtain the theoretical emf's given below:

<u>H₂</u>	<u>H₂O</u>	<u>E(theoretical), mV</u>	<u>E(actual) mV</u>
113	2.08	1131	1094
390	2.22	1183	1148
950	1.88	1229	1185

To a good approximation, the actual cell output, within the range of the current tests, may be represented by the expression:

$$E(\text{actual}), \text{ mV} = 933 + 94.1 \log (H_2/H_2O)$$

This empirical expression is only one possible means of interpreting the data. It does not take into account the apparent O₂ inleakage to the cell. An apparent leak becomes a major factor when the oxygen level, in equilibrium with the H₂/H₂O ratio, becomes small, and a significant portion of the free O₂ in the cell is due to the "leak." The cell does exhibit an apparent leak and this limits the validity of an extrapolation of the empirical expression beyond a certain bound. With this in mind, the empirical expression may be used to investigate the behavior of the detector at or near the range in which the experimental data were obtained. For example, the system has been set-up so that an O₂ analyzer output of 1000 mV will activate the back-up gas system. According to the above expression, the H₂/H₂O ratio at that point is 5.15. It would require 31.7 μ atm of O₂ in the loop to reduce the H₂ and increase the H₂O levels to this ratio.

At the other end of the scale, it is possible to predict the drop in oxygen cell reading to be expected for the minimum amount of oxygen detectable by the gas chromatograph. A peak of 0.05 mV at a typical sensitivity of 3 μ -atm/mV would indicate the presence of 0.15 μ -atm O₂ in the loop. This would react with H₂ in the O₂ cell to form 0.30 μ -atm H₂O, reducing the H₂/H₂O ratio from its nominal 200 to 173.8 and decreasing the cell output from 1149 to 1144 mV.

A new version of Applied Electrochemistry's O₂ analyzer cell, which was designed for use of a reference gas other than air, was in the loop during the calibration tests discussed above. This cell had exhibited lower leak rates during leak checks than the conventional cells, and it was anticipated that this cell would yield readings closer to the theoretical values. Apparently, there is a major design flaw with this unit, since it gave a nearly constant output of 870 mV during all the calibration tests. This cell has been removed and returned to the vendor.

2.5 TASK 5 - MECHANICAL PROPERTIES EVALUATION

2.5.1 Test Specimen Machining

Machining of the test specimens required for the screening test program was completed by September, 1978.

2.5.2 Selection of Stresses for Screening Creep Tests

Based on the 300-hour creep and rupture test results, stresses were selected for the following three types of screening creep tests:

1. Stress for 1% total strain in 100,000 hours; to be tested for 10,000 hours.
2. Stress for 1% total strain in 6,000 hours; to be tested for 6,000 hours.
3. 80% of the stress for rupture in 10,000 hours; to be tested for 3,000 hours.

During the screening tests, the amount of creep on each sample will be measured at 1,000-hour intervals, following which the samples will be rehung at the same load unless excessive creep is occurring. In that case, the specimen will be removed from test and replaced in the stringer by a new specimen.

The selection of desired stresses for all DCHT alloys for screening creep testing was completed and reported in the Quarterly Report for July 1, 1978 - September 30, 1978, report number C00-2975-25.

The selection of desired stresses for the NPH alloys was reported in the Quarterly Report for January 1, 1979 to March 31, 1979, report number C00-2975-31.

Based on the desired stress levels for each alloy, loading schedules for screening (multi-specimen) creep testing have been prepared. The multi-specimen creep (MSC) tests are performed on stringers of specimens with up to fifteen (15) specimen per stringer. There are six stringers for each MSC stand, and one stand will be used for the testing at each of the exposure temperatures, 750^o, 850^o, 950^o, and 1050^oC. Two sizes of specimens are used for these tests (gauge diameters of 6.40 and 9.07 mm (0.252 and 0.357 inches)). One size or the other was used for a particular alloy based on the alloy's strength, the larger specimen being used for the weaker alloys. Since only one load can be applied to each stringer, there are only two possible stress levels in a stringer, one for each specimen size. From the desired stress levels, specimens were segregated into six stringer groups per retort, i.e. per exposure temperature.

Each group of specimens was chosen so that the load in their particular stringer would result in the closest approximation of, without exceeding, the desired stresses for each alloy in the group. The actual specimen stresses, the test conditions, and the desired (calculated) stresses are listed in Tables 2.7 and 2.8 for the NPH and DCHT alloys, respectively.

In addition to the specimens described above, nine (9) TZM specimens are being tested in single specimen (precision) creep tests for the screening creep program. The inordinately high strength of the alloy precluded the possibility that TZM specimens could be stressed at significant levels in the MSC tests, where the maximum loadings are limited by the weaker alloys. The test conditions for the separate TZM tests are shown in Table 2.9.

During this quarter, four (4) additional TZM tests were brought on line. This brings the total number of TZM screening creep tests to six (6) presently on line. The status of these tests also is shown in Table 2.9. The remaining three (3) TZM tests will be brought on line next quarter.

2.6 TASKS 6 AND 7 - THERMAL STABILITY EVALUATION AND PRIMARY COOLANT CORROSION RESISTANCE EVALUATION

Four (4) Aging Corrosion (A/C) retorts were brought on line during this quarter. These include specimens for exposure for 1000, 3000, 6000, and 10,000 hours at 750^o and 850^oC, (1382^o and 1562^oF). The specimens include structural stability pins for examination at 1000, 3000, 6000, and 10,000 hours, impact specimens for post-exposure testing after 3000 and 10,000 hours, and tensile specimens for post-exposure testing after 3000, 6000, and 10,000 hours. Thus, 66 tensile specimens, 22 impact specimens, 167 structural stability pins and 26 weldments are on test at 750^oC (1382^oF), and 76 tensile specimens, 26 impact specimens, 167 structural stability pins and 26 weldments at 850^oC (1562^oF), for a total of 572 specimens on test. The exact test plan, including specific testing for each alloy, was presented in the quarterly report for July 1, 1977 to September 30, 1977, report number C00-2975-17. The test status for the four retorts presently on line is shown in Table 2.10. The other four (4) retorts for exposures at 950^o and 1050^oC will be brought on line at the beginning of the next quarter.

TABLE 2.7

**DESIRED (CALCULATED) AND ACTUAL STRESS LEVELS
FOR NPH ALLOY SCREENING CREEP SPECIMENS**

Alloy	Temperature °C	3000 Hours at 80% Stress for Rupture in 10000 Hours				6000 Hours at 100% Stress for 1% Total Strain in 6000 Hours				10,000 Hours at 100% Stress for 1% Total Strain in 100000 Hours			
		Desired		Actual		Desired		Actual		Desired		Actual	
		MPa	KSI	MPa	KSI	MPa	KSI	MPa	KSI	MPa	KSI	MPa	KSI
HK-40	750	62	9	62	9	69	10	62	9	48	7	48	7
	850	34	5	34	5	34	5	28	4	21	3	21	3
	950	14	2	14	2	14	2	14	2	7	1	6	0.9
	1050	7	1	7	1	6	0.9	3	0.5	3	0.4	2	0.3
IN-519	750	69	10	62	9	76	11	76	11	55	8	48	7
	850	34	5	34	5	34	5	28	4	28	4	28	4
	950	14	2	14	2	21	3	17	2.5	7	1	6	0.9
	1050	6	0.8	3	0.5	7	1	7	1	2	0.3	2	0.3
Hastelloy X	750	69	10	62	9	55	8	48	7	34	5	21	3
	850	28	4	28	4	28	4	21	3	14	2	7	1
	950	14	2	14	2	7	1	6	0.9	4	0.6	0.7	0.1
	1050	3	0.5	3	0.5	3	0.4	2	0.3	0.7	0.1	0.3	0.05
Inconel 617	750	96	14	76	11	90	13	76	11	62	9	62	9
	850	48	7	34	5	41	6	34	5	28	4	28	4
	950	21	3	17	2.5	21	3	17	2.5	14	2	14	2
	1050	7	1	7	1	7	1	7	1	3	0.4	2	0.3
Inconel 617- Coated	750	76	11	76	11	55	8	48	7	34	5	21	3
	850	34	5	34	5	28	4	21	3	14	2	7	1
	950	14	2	14	2	14	2	9	1.25	6	0.8	0.7	0.1
	1050	7	1	3	0.5	3	0.5	2	0.3	1.4	0.2	0.3	0.05
Mo-Re 2	750	90	13	76	11	76	11	76	11	55	8	48	7
	850	48	7	34	5	41	6	34	5	28	4	28	4
	950	21	3	17	2.5	21	3	17	2.5	14	2	14	2
	1050	7	1	7	1	7	1	7	1	6	0.8	3	0.5
MA-956E	850	55	8	55	8	55	8	41	6	48	7	41	6
	950	48	7	48	7	48	7	34	5	41	6	34	5
	1050	41	6	21	3	41	6	21	3	34	5	21	3
Alloy 800H	750	48	7	48	7	48	7	48	7	34	5	21	3
	850	14	2	7	1	21	3	21	3	14	2	7	1
	950	7	1	6	0.9	7	1	6	0.9	2	0.3	0.7	0.1
Alloy 800H- Coated	750	41	6	21	3	28	4	21	3	21	3	21	3
	850	14	2	7	1	14	2	7	1	7	1	7	1
	950	7	1	6	0.9	3	0.4	0.7	0.1	0.7	0.1	0.7	0.1
Incoloy 802	750	90	13	76	11	83	12	76	11	55	8	48	7
	850	41	6	34	5	41	6	34	5	21	3	21	3
	950	14	2	14	2	14	2	9	1.25	7	1	6	0.9
Incoloy 807	750	69	10	62	9	62	9	62	9	41	6	21	3
	850	28	4	28	4	28	4	21	3	14	2	7	1
	950	14	2	14	2	7	1	6	0.9	4	0.6	0.7	0.1
	1050	5	0.7	3	0.5	3	0.4	2	0.3	0.7	0.1	0.3	0.05
Incoloy DS	750	34	5	21	3	34	5	21	3	21	3	21	3
	850	14	2	7	1	14	2	7	1	14	2	7	1
	950	7	1	6	0.9	5	0.7	0.7	0.1	3	0.4	0.7	0.1
HD-556	750	96	14	76	11	62	9	62	9	41	6	21	3
	850	48	7	34	5	28	4	21	3	21	3	21	3
	950	21	3	17	2.5	14	2	9	1.25	6	0.9	6	0.9
	1050	7	1	7	1	4	0.6	3	0.5	1.4	0.2	0.3	0.05
Maurite 36XS	750	55	8	48	7	55	8	48	7	41	6	21	3
	850	34	5	34	5	34	5	28	4	21	3	21	3
	950	14	2	14	2	14	2	14	2	7	1	6	0.9
	1050	7	1	7	1	6	0.9	3	0.5	3	0.4	2	0.3

TABLE 2.8

DESIRED (CALCULATED) AND ACTUAL STRESS LEVELS
FOR DCHT ALLOY SCREENING CREEP SPECIMENS

Alloy	Temperature °C	3000 Hours at 80% Stress for Rupture in 10000 Hours				6000 Hours at 100% Stress for 1% Total Strain in 6000 Hours				10000 Hours at 100% Stress for 1% Total Strain in 10000 Hours			
		Desired		Actual		Desired		Actual		Desired		Actual	
		MPa	KSI	MPa	KSI	MPa	KSI	MPa	KSI	MPa	KSI	MPa	KSI
TZM ^(a)	850	351	51	351	51	386	56	386	56	324	47	324	47
	950	282	41	282	41	296	43	296	43	234	34	234	34
	1050	193	28	193	28	186	27	186	27	124	18	124	18
TZM	750	400	58	345	50	482	70	345	50	427	62	345	50
	850	351	51	152	22	386	56	152	22	324	47	152	22
	950	282	41	48	7	296	43	48	7	234	34	48	7
TZM (Coated)	1050	193	28	76	11	186	27	76	11	124	18	76	11
	1050	193	28	76	11	186	27	76	11	124	18	76	11
MA-754	850	124	18	110	16	138	20	110	16	131	19	110	16
	950	96	14	48	7	117	17	48	7	103	15	48	7
	1050	69	10	21	3	90	13	76	11	76	11	76	11
MM-0011 CC	750	400	58	345	50	386	56	345	50	276	40	262	38
	850	186	27	152	22	200	29	152	22	138	20	110	16
	950	69	10	48	7	83	12	48	7	45	6.5	34	5
MM-0011 DS	1050	14	2	14	2	14	2	14	2	10	1.5	7	1
	750	420	61	345	50	455	66	345	50	345	50	345	50
	850	193	28	152	22	248	36	152	22	179	26	152	22
RENE' 100	950	76	11	48	7	110	16	48	7	62	9	48	7
	1050	21	3	21	3	28	4	21	3	21	3	21	3
	750	345	50	345	50	386	56	345	50	269	39	262	38
RENE' 80	850	152	22	152	22	179	26	152	22	110	16	110	16
	950	34	5	34	5	55	8	48	7	24	3.5	17	2.5
	1050	7	1	7	1	7	1	7	1	3	0.5	0.7	0.1
IN-738	750	345	50	345	50	345	50	345	50	262	38	262	38
	850	152	22	152	22	186	27	152	22	124	18	110	16
	950	34	5	34	5	62	9	48	7	24	3.5	17	2.5
Alloy 713 LC	750	296	43	262	38	331	48	262	38	241	35	152	22
	850	131	19	110	16	117	17	110	16	62	9	55	8
	950	21	3	17	2.5	21	3	17	2.5	7	1	1.4	0.2
NX-188	1050	1.4	0.2	0.7	0.1	1.4	0.2	0.7	0.1	0.7	0.1	0.7	0.1
	750	303	44	262	38	365	53	345	50	262	38	262	38
	850	138	20	110	16	124	18	110	16	62	9	55	8
Udimet 720	950	24	3.5	17	2.5	21	3	17	2.5	7	1	1.4	0.2
	750	303	44	262	38	365	53	262	38	262	38	262	38
	850	138	20	110	16	152	22	110	16	83	12	69	10
	950	24	3.5	17	2.5	34	5	28	4	21	3	17	2.5
	750	303	44	262	38	276	40	262	38	200	29	152	22
	850	124	18	110	16	110	16	110	16	55	8	55	8
	950	17	2.5	17	2.5	17	2.5	17	2.5	7	1	1.4	0.2

TABLE 2.9

TEST CONDITIONS AND STATUS OF TZM
SCREENING CREEP TESTS IN PRECISION,
SINGLE SPECIMEN CREEP EQUIPMENT

Test Stand	Test Conditions		Stress (MPa)	Status Hours on Test
	Test Temp. (°C)	Test Duration (hrs.)		
ST-1	950	10,000	234	525
ST-2	1050	10,000	124	377
ST-3	1050	3,000	193	0
ST-5	850	3,000	351	669
ST-6	950	6,000	296	256
ST-7	1050	6,000	186	0
ST-8	950	3,000	282	0
ST-9	850	6,000	386	620
ST-10	850	10,000	324	477

TABLE 2.10

TEST CONDITIONS AND STATUS OF AGING-
CORROSION TESTS

Test Stand	<u>Test Conditions</u>		Test Status (Hours on Test)
	Test Temp. (°C)	Test Duration (hrs.)	
AC-1	950	6,000 & 10,000	0
AC-2	950	1,000 & 3,000	0
AC-3	850	6,000 & 10,000	207
AC-4	850	1,000 & 3,000	325
AC-5	750	6,000 & 10,000	485
AC-6	750	1,000 & 3,000	551
AC-7	1050	6,000 & 10,000	0
AC-8	1050	1,000 & 3,000	0

2.7 TASK 9 - DATA MANAGEMENT AND PROGRAM MANAGEMENT

2. .1 Data Management - (R.B. Hand)

2.7.1 Data Storage Procedures

Modification of the data storage computer program is progressing. The changes will provide for encoding compound compositions, gas flow rates, automatic calculation and encoding of time weighted averages and standard deviation for gas chemistries, flow rates and temperatures of test environments, future expansion and improved material identification and traceability.

The program to initialize the file is complete.

2.7.1.2 Data Retrieval Procedures

Concurrent with the issuing of the revised instruction manual in July, simple card image retrievals based on material identity numbers (MINO), data sets, and specified materials properties will be possible; 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 data set (Material Description and Test Results, Specimen Data, Test Planning and Specimen Location, etc.) or a particular property (tensile, creep, fatigue, etc.) 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, vendor, test parameters, etc. Development of these procedures will require a major effort and time expenditure.

2.7.2 Program Management

A meeting was held with J.E. Fox, Program Technical Monitor, and T. Werner, HTGR Section Chief, on April 4, 1979, to review the program status and to recommend changes in the Base Program Plan.

A meeting was held with all Schenectady area program contributors on April 9, 1979, for a general program review.

Weekly review meetings were held with the High Temperature Reactor Materials Testing Laboratory (HTRMTL) personnel to facilitate the restart of loop operation during the shutdown period in May and June, 1979. Meetings were held monthly and, at times, more frequently with all Schenectady program personnel. A review meeting with program personnel in Evendale, Ohio was held on April 26, 1979. This review covered work activities on the Direct Cycle Helium Turbine (DCHT) alloys and the data management task.

Representatives from the French Commissariat a L'Energie Atomique visited General Electric, Schenectady on June 21, 1979 for the purpose of technical/program information exchange.

The Program Master Schedule is presented in Figure 2.1. This schedule is a revision and reflects schedule changes that are primarily due to the start of the screening test program during the last quarter, January to March, 1979.

FIGURE 2.1 PROGRAM MASTER SCHEDULE

Section 3.0

WORK TO BE COMPLETED DURING THE NEXT QUARTER

3.0 WORK TO BE COMPLETED NEXT QUARTER

- Aging-corrosion exposures will be started at 950°C and 1050°C.
- The 1000-hour aging-corrosion exposures will be completed at 750°C, 850°C, 950°C, and 1050°C. The post exposure analysis on the metallography/weight gain, tensile and impact specimens will commence.
- The final TZM screening creep tests in the single specimen creep equipment will be started.
- Two multi-specimen creep stands, one at 750°C and one at 850°C will be started on test.
- Installation of Loop No. 2 will be continued.
- Alloy selection for the intensive screening testing phase of the program will be finalized for approval.