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ADVANCED GAS COOLED NUCLEAR REACTOR MATERIALS EVALUATION AND DEVELOPMENT PROGRAM

PROGRESS REPORT
FOR PERIOD

July 1, 1979 - September 30, 1979

BY
PROJECT STAFF

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

GENERAL ELECTRIC

March 7, 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 Principal 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. Major contributors to the Program are:

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ABSTRACT

This report presents the results of work performed from July 1, 1979 through September 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
April 1, 1979 to June 30, 1979	C00-2975-34

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

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

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.

2.2.2.3 Materials Joining Studies

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

2.3 TASK 3 - TESTING AND ANALYSIS FACILITIES AND EQUIPMENT

2.3.1 Multi-Specimen Creep (MSC) Equipment

The second multi-specimen creep (MSC) stand, scheduled for use at 850°C (1562°F), was calibrated during the report period. One MSC stand being utilized at 750°C (1382°F) had already been calibrated. Both of these stands have Inconel 601 retorts and a 99.1-cm (39-inch) long hot zone. The calibration is performed with all six pull rods and load arms in place and loaded to their approximate service loads. The calibration is then accomplished by varying the loads above and below the specified service loads. This method is used because of the possible interaction between the six load trains in a given retort under load.

Screening creep testing was initiated in these stands during the quarter. A total of seventy-four (74) specimens are on test at 750°C (1382°F), and seventy-three (73) specimens at 850°C (1562°F).

The load train travel on the MSC stands presents an experimental problem. Because of the design of the test stands, there is insufficient room for lever arm movement to allow for contraction during cooling of the assembly under load to room temperature from the test temperature. The 1:10 weight pan to specimen load ratio and the 99.1 mm (39-inch) active stringer length means that the lever arm movement due to thermal contraction of specimens under cooling is so large that the arm movement is restricted. This situation presents a problem once the test is underway, since each stand must be cooled to room temperature, disassembled, and the specimens measured every 1000 hours. (For the start of the test, the specimens are loaded after they reach test temperature.) In order to maintain all anelastic strain and to prevent specimen annealing in an unstressed

state, one would prefer to cool the specimens as far as possible under load, ideally all the way to room temperature. The present system allows for cooling at least 100°C (180°F) below the test temperature before specimens are unloaded. It is felt that while this is not ideal, the loss of the anelastic strain component due to this limitation is below the resolution capabilities of the strain measuring device, ± 0.00127 mm (± 0.00005 inches). A review of the test stand design is now being made to determine if it is possible to modify the stand to accommodate the amount of lever arm movement or uptake required to cool all the way to room temperature under load.

2.3.2 Single Specimen Creep-Rupture (ST) Equipment

One of the ST stand furnace windings burned out after approximately 2000 hours of operation. The design life of these elements is 20,000 hours. It is not yet clear whether this is an isolated incident, or due to the number of thermal cycles a few of the furnaces have been put through for check-out and in service. One preventative measure that will be taken is to keep the furnaces at some level of power at all times. The furnaces will be supplied enough power to maintain a temperature of 93°C (200°F) when off test. This will lessen the thermal shock to the windings when they are cycled to temperature, and has the added benefit that it reduces H₂O ingress into the furnace ceramic when idle.

2.3.3 Fatigue Test Equipment

Approval for the purchase of two environmental test chambers for conducting fatigue testing in controlled purity He was given by DOE-Chicago Operations Office. A purchase order was placed with Centorr Associates, Suncook, N.H.

A design for the independent controlled purity He system for fatigue testing was completed and is presently undergoing a design review. It is necessary to design and build a separate He supply subsystem for the fatigue test stands because of the large volume of the retorts, 90 ℓ, and the frequency of opening retorts to air to start new tests. The burden of maintaining a He test atmosphere within reasonable limits in either of the two main He loops with the fatigue system connected to them was thought to be too great a risk. The volume of either loop is approximately 240 ℓ, so the addition of 90 ℓ or 180 ℓ from one or both fatigue retorts up to several times a week is judged to be impractical.

The fatigue He supply facility will be a once-through-then-out (OTTO) system, with controlled (metered) impurity gas species added to purified He. A complete layout of this system will be shown in the report for the next quarter.

2.3.4 Temperature Control System

A problem with the computerized temperature control system has been identified. Short interruptions in laboratory power are handled by a power back-up system which is activated automatically upon loss of the main power supply. This switch-over is adequate to maintain operation of all laboratory components except the computerized temperature control system. Even the shortest interruptions in power perturbs the computer sequencing operation, requiring the temperature controlling program to be reinitialized. In order to circumvent this problem, a continuous battery-based power supply will be installed to provide an uninterruptable power source. The battery system will be connected to the laboratory power for continuous charging. In the case of power interruption, the battery system will supply continuous power to the computer during back-up power switchover. This battery power supply system will be provided by General Electric at no cost to the contract.

2.4 TASK 4 - SIMULATED REACTOR HELIUM ENVIRONMENT

2.4.1 Loop No. 1 Operation

The control of loop chemistry in Loop No. 1 was satisfactory over the report period. Table 2.1 compares the average loop chemistry and the standard deviations for the period with the target chemistry and ranges for the loop. There were a few minor excursions out of the set ranges during the period, mostly due to chemistry control during retort or test start and tie-in to the loop. These excursions last usually an hour or less and have been confined to minor deviations across the permissible upper or lower range gas composition boundary.

It was observed that methane cracking, $\text{CH}_4 \rightarrow \text{C} + 2\text{H}_2$, was significant when retorts operating at 1050°C (1922°F) were on line.

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

Helium path revisions have been made in the back-up, purge, and fill gas systems. In the original design, back-up gas (He with 500 ppm H_2 and < 3 ppm H_2O)

TABLE 2.1

LOOP NO. 1 GAS CHEMISTRY HISTORY
JULY 1, 1979 THROUGH SEPTEMBER 30, 1979

<u>Constituent</u>	<u>Target Chemistry (μ atm)</u>	<u>Actual Average Chemistry (μ atm)</u>	<u>Standard Deviation (μ atm)</u>
H ₂	400	405	22
H ₂ O	2.0	1.9	0.2
CO	40	39	2
CO ₂	0.2	0.2	0.01
CH ₄	20	18	1.5
N ₂	6	6.6	2.6

was to be purified by passage over hot Ridox, essentially a metal catalyst favoring the reaction of any oxygen present, either to form a metal oxide or water vapor. Fill gas was to be commercially pure helium purified by passage through a liquid nitrogen cooled activated charcoal trap (LN_2 --ACT). Purge gas was to be the same composition as back-up gas, but purified by passage through a LN_2 --ACT.

As experience was gained with system operating characteristics, especially with regard to loop impurity level changes associated with tying-in of test retorts containing purge gas, it became evident that fill, back-up, and purge gases could, and should, all be of the same composition and be subjected to essentially the same purification sequence; oxygen removal in each case, plus water removal in the case of fill and purge gases. Accordingly, a design was devised whereby helium containing the desired 500 ppm H_2 was bled from a double manifold which permitted four, individually replaceable bottles of pre-mixed gas to be used first, plus up to 16 bottles, replaceable in groups of eight, to be available in the event of back-up gas system activation. The final revision to the design was to use two separate Ridox canisters so that one serves the back-up system only and the second serves the purge and fill systems. This separation permitted the two systems to operate at different pressures as desired and eliminated a check valve cross-leakage problem which had caused a tendency for overpressurization of the back-up system and loop gas into the purge system. All plumbing revisions have been completed and leak checked and the Ridox No. 1 canister (in the back-up system) is being regenerated. Ridox No. 2 was previously regenerated and is serving the purge and fill systems. A molecular sieve has been installed downstream of the Ridox No. 2 to provide the desired moisture level control on purge and fill gases.

Also completed during the quarter was the installation of an automatic fill/dump system designed to maintain return receiver helium pressure near its desired level of 5 psig. A pressure switch with high and low set points activates a solenoid valve to a vent if receiver pressure reaches 6.5 psig and a solenoid valve in the fill line if pressure drops to 4.5 psig. Each line contains a calibrated rotameter, a preset flow regulating valve, and an elapsed time meter recording the total time each system is operating. Daily readings of these times will be made to allow tracking of the helium inventory and alert the loop operator to developing system problems. A small leak, for example, may not be enough to perturb the gas chemistry within the limits of measurement accuracy, but will show up as unusual activity of the fill system.

The helium system replumbing necessary for installation of an automatic LN₂ filling system for the main LN₂-ACT dewar has been completed. This involved relocating the LN₂-ACT remote from its original site in the PP&A. To accomplish this without interfering with the operation of Loop No. 1, a separate LN₂-ACT was installed at the desired new location. Plumbing connections were made to a flowmeter and shutoff valves. When the newly installed LN₂-ACT has been leak checked and regenerated, it will become the primary unit for PP&A No. 1. The present main LN₂-ACT will then be removed from its site in PP&A No. 1, moved to its new location (adjacent to the PP&A No. 1 primary unit discussed above) and plumbed into PP&A No. 2 as the primary unit for Loop No. 2. Finally, both main LN₂ dewars will be connected to an automatic solenoid valve device to maintain LN₂ within desired levels. At present the dewar must be manually filled twice daily.

Over-temperature controllers (OTC's) have been connected to all the heaters devoted to gas purification functions in PP&A No. 1 and the back-up, fill, and purge gas systems. Specifically, five heaters are involved; two Cu canisters for O₂ control, one CuO canister for H₂ and CO removal, and two Ridox canisters, one for back-up gas and one for purge and fill systems. Appropriate provisions have been made for addition of OTC's required for the Cu and CuO canisters in PP&A No. 2. Also installed was a central readout station for thermocouples attached to each of these heated canisters in order to facilitate routine status checks.

Finally, an alarm system has been installed to provide a signal to a constantly monitored display board in the Plant Security offices. This alarm is activated for any of the following conditions which could develop in the laboratory or Loop No. 1:

1. Low helium pressure in loop.
2. Low discharge pressure, any helium pump.
3. High moisture level in loop gas.
4. High oxygen level in loop gas.
5. Back-up gas system activated.
6. Room temperature high.
7. Cooling water temperature high.
8. Stand-by water pump activated.
9. Domestic cooling water activated.
10. Temperature control system crash.
11. Test retort temperature high or low.

2.4.3 Pumping, Purification and Analysis Subsystem (PP&A) No. 2

During the report period installation of several modifications to PP&A No. 2 was completed. These changes were all to improve system performance and have been proven in Loop No. 1. Principal items include:

1. Modification of impurity gas addition valves to permit rapid pick-up of metered additions and facilitate mixing into the main helium charge.
2. Replumbing of the eight gas addition valves to operate in separable banks of four to allow valve cleaning, repair, etc. without loop shutdown.
3. Installation of shut-off and bypass valving for the 16-port sense line selector valve to permit valve service or replacement without system shutdown.
4. Replumbing the impurity addition flow path such that a sample of the helium flow used for rapid pick-up of material impurities can be routed to the gas analysis station.
5. Welding of several previously threaded pipe joints in the main one inch diameter helium supply line.

In addition, revisions to the seals on the helium pumps for PP&A No. 2 were designed and the necessary machining work initiated. Original construction of these bellows pumps falls far short of the leak tightness felt to be needed for this particular application.

Finally, leak checking operations on PP&A No. 2 were begun. Initial effort has been in the instrumentation gas connections to permit preliminary checkout of instrumentation to proceed in parallel with leak checking operations on the remainder of the gas system.

2.4.4 Instrumentation

A new carrier gas trap has been fabricated and installed. The original version had some effective trapping capacity above the minimum LN_2 level normally maintained. Consequently, there was a noticeable baseline shift on the gas

chromatograph associated with the LN_2 level in the carrier gas trap. The new version has all its effective trapping capacity submerged in LN_2 at all times and has eliminated the baseline shift problem.

Oxygen analyzer degradation remains a problem. This instrument is based on electrochemical principles; the emf developed at opposite surfaces of a zirconia tube by virtue of different oxygen concentrations is measured. In the particular instrument employed, the outside oxygen level in air is used as a constant value while the remaining oxygen in loop helium after equilibrium at 750°C (1382°F) within the zirconia tube completes the electrochemical cell. The problem is that the developed emf decays with time ($.5\text{--}1\text{ mV/day}$) which would indicate a free oxygen increase in the loop helium if interpreted at face value. It is observed that this decay proceeds even when the impurity levels in the loop are maintained constant, as measured by the gas chromatograph and hygrometers.

At present, it is believed that the problem is associated with degradation of the cell in the presence of the particularly low oxidation potentials of the loop helium at high temperatures. An interim procedure implemented to minimize the problem is to periodically "regenerate" the existing cell by a high temperature $\{850^\circ\text{C} (1562^\circ\text{F})\}$ exposure to pure oxygen. This procedure is recommended by the cell vendor. This procedure can be performed in-house without disturbing the cell or interfering greatly with loop operation. Although the cell output continues to degrade slowly, it is still regarded as the quickest indicator of leakage problems in the loop. A permanent solution to this situation is presently under investigation.

The EG&G Dew Point Hygrometer has developed electronic problems and will be returned to the vendor for warranty repairs. The unit had been purchased and installed in series with the Shaw hygrometer probe (which detects moisture level changes by measuring the capacitance of a thin alumina layer) to serve as a monitor and calibration standard. The Shaw unit will continue to be used until the EG&G can be repaired, re-calibrated, and re-installed in the loop. Data obtained by the Shaw unit over this time period will be corrected if it is shown, by measurements made with the EG&G, that any drift has occurred in the Shaw units' calibration.

Gas line connections to GC No. 2 have been completed and leak checked. A revision to the seals on one of the two ionization detector housings was completed to conform to the previous change made on GC No. 1

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 Stress for Screening Creep Tests

Stresses for multispecimen creep (MSC) testing in the screening phase of the program have been selected and were reported in the quarterly report for the period April 1, 1979 to June 30, 1979, report number C00-2975-34.

2.5.3 Multispecimen Creep Testing

Multispecimen creep tests at 750°C (1382°F) and 850°C (1562°F) were initiated during the quarter. The 750°C (1382°F) test had reached the first one thousand (1000) hour test interval in September 1979, at which time the test was interrupted and the specimen stringers disassembled. The procedure is as follows: the furnace is cooled at least 100°C (180°F) below test temperature and all six (6) lever arms are unloaded simultaneously. The test stand is allowed to cool to room temperature under loop He; then the retort is segregated from the loop. The retort is opened, the stringers removed and the specimens' surface appearances are characterized by visual examination and color photography. The stringers are disassembled and length measurements are made with a special digital measuring device which measures the distance between specimen shoulder grooves. The accuracy of the measurements is ± 0.00127 mm (± 0.00005 inches). Then the total plastic strain is calculated using these measurements and the pre-exposure length measurements.

The creep strains are reviewed before the specimens are re-assembled into stringers so that specimens showing abnormally high creep strains can be taken off test and replaced. However, the test conditions are such that rupture should not occur in any specimen.

The test restart procedure is essentially the inverse of the shutdown procedure, except that the test stand is subjected to several purge and evacuation cycles with purge gas (He with 500 ppm H_2 and < 3.0 ppm H_2O) at room temperature and $204^{\circ}C$ ($400^{\circ}F$) before being filled with loop He and brought to test temperature. The loads are applied simultaneously after the specimens reach the temperature at which the loads were removed during the shutdown procedure; at least $100^{\circ}C$ ($180^{\circ}F$) below test temperature.

Along with the screening creep testing in the MSC stands, nine (9) complimentary screening creep tests with TZM alloy are scheduled for the precision creep (ST) stands. At the end of the quarter, eight (8) of the nine (9) TZM specimens to be tested in the single specimen creep stands were running. This is an addition of two (2) tests to the six (6) that were on line at the end of the last quarter.

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

The structural stability pins, weldments, tensile specimens and impact bars are to be exposed at 750° , 850° , 950° , and $1050^{\circ}C$ (1382° , 1562° , 1742° and $1922^{\circ}F$) for intervals of 1000, 3000, 6000 or 10,000 hours. The exact test matrix is presented in the quarterly report for July 1, 1977 to September 1, 1977, report number C00-2975-17. There are eight (8) test retorts for these exposures. Two of these aging/corrosion (A/C) stands are used at each of the four temperatures and the specimens are segregated into two (2) groups for test, one for 1000 and 3000 hours and another for 6000 and 10,000 hours. The test exposures are interrupted at 1000 or 6000 hours, respectively, and the appropriate specimens unloaded. The other specimens are left in the retorts and are exposed for the additional test time to reach their scheduled exposure.

The $750^{\circ}C$, 1000- and 3000-hr exposure stand (A/C-6) reached 1,000 hrs of exposure time during mid-July. The 1000-hr structural stability pins and weldments were removed for post-exposure evaluation. The structural stability pins have been weighed after removal from the furnace, photographed, the surface condition of the specimens documented, and the pins have been sectioned and mounted for metallography.

On August 1, the 850°C/1000- and 3000-hr aging and corrosion stand (A/C-4) reached 1000 hrs of exposure time and was shut down to remove the 1000-hr specimens. Upon opening the retort, it was found that 28 structural stability pins in and near furnace Zone 4 had fallen from the ceramic supports due to failure of the 0.41mm (0.016 in.) nichrome hanging wires. Zone 4 had experienced an over-temperature of 221°C (398°F) on July 16, 1979, subsequent to an incorrect temperature control computer entry after a power failure. (The over-temperature problem was discovered about 0.5 hr after the entry was typed into the computer and the total excursion time amounted to about 1.5 hrs. Approximately 33 NPH specimens were involved in the over-temperature to some extent.)

Based on the findings from A/C-4, (the 850°C/1000- and 3000-hr aging and corrosion stand) both 1050°C A/C retorts, both 950°C A/C retorts, and the remaining 850°C A/C retort were shut down for inspection. (The two 750°C retorts were also subsequently shut down). All structural stability pins had fallen off the ceramic support posts at 950°C and 1050°C. In addition, four tensile, one impact and two weldment specimens had fallen in A/C-8 (1050°C/1000- and 3000-hr); five tensile, two impact and four weldment specimens had fallen in A/C-7 (1050°C/6000- and 10,000-hrs). Note that the mechanical test specimens and weldment coupons were hung with Inconel 601 0.89 mm (0.035 in.) dia. wire. All specimens in the 850°C/6000- and 10,000-hr furnace (A/C-3) were still in place, as were the samples in both of the 750°C retorts. This information is summarized in Table 2.2.

Inspection of Table 2.2 reveals that approximately 219 structural stability pins were found in the A/C retort end caps. Since the specimens had fallen after an indeterminate period of exposure, and since the thermal and atmospheric conditions in the end caps are unknown, the exposure conditions of these specimens cannot be accurately determined.

Visual observations of the failed nichrome wire in A/C-7 (1050°C/6000- and 10,000-hrs) revealed a liquated condition at the fracture location in a number of cases. Representative fracture surfaces of both varieties of wire were also examined under the scanning electron microscope (SEM). Energy dispersive analysis by x-ray (EDAX) of the fracture surfaces indicated the presence of trace amounts of phosphorus. Additionally, Cr-rich particles were observed on the fracture surface of the nichrome wire and Al-rich particles were in evidence on the fracture surface of the Inconel 601 wire.

TABLE 2.2

AGING/CORROSION (A/C) TEST SPECIMEN STATUS

<u>Retort</u>	<u>Temp. & Exposure Time</u>		<u>Hrs. On Test</u>	<u>Structural Stability</u>		<u>Mechanical Test</u>	
				<u>Pins</u>		<u>Specimens & Welds</u>	
				<u>In Test</u>	<u>Fallen</u>	<u>In Test</u>	<u>Fallen</u>
A/C-6	750°C	1,000 & 3,000 hrs.	1279	82	None	46	None
A/C-5	750°C	6,000 & 10,000 hrs.	1575	85	None	68	None
A/C-4	850°C	1,000 & 3,000 hrs. (a)	1000	82	28	50	None
A/C-3	850°C	6,000 & 10,000 hrs.	1104	85	None	75	None
A/C-2	950°C	1,000 & 3,000 hrs.	629	82	All (66) (b)	50	None
A/C-1	950°C	6,000 & 10,000 hrs.	550	85	All (28)	75	None
A/C-8	1050°C	1,000 & 3,000 hrs. (a)	585	82	All (59)	40	7
A/C-7	1050°C	6,000 & 10,000 hrs.	479	85	All (66)	53	11

NOTES:

(a) Retort experienced overtemperature

(b) () Indicates number of specimens found in end cap

It was observed that the nichrome wires failed usually in areas adjacent to the Sauereisen #8 ceramic cement which had been used to hold them in place. Because of this, it was suspected that some agent in the cement may have contributed to the failures. The vendor, who had, at the outset of the program, claimed that the cement would be suitable for use in the He test atmosphere, was contacted again for the constituents of the cement. The cement was found to contain mono-magnesium phosphate, magnesium oxide and zirconium silicate. An excess of mono-magnesium phosphate, beyond that needed for the cementation process, is contained in the base powder to assure adequate availability of that material. Of the agents available, phosphorus was identified to be potentially the most deleterious, if released, because of the low melting point eutectic it can form with Ni.

Determination of the phosphorus content in the cement in the as-received condition and after exposure in controlled-purity helium at the four test temperatures indicated that the phosphorus level remaining in the cement decreased with increasing exposure temperature (see Table 2.3). The phosphorus levels also suggest that the element was released from the mono-magnesium phosphate which had taken part in the cementation reaction as well as from the excess present. This indicates that other phosphorus compounds in the cured cement are unstable in this environment as well. To determine if any other undesirable elements were present in the ceramic cement, a complete qualitative spectrographic analysis of the as-received powder was performed. These results are shown in Table 2.3 and indicate the presence of a number of elements in "trace" quantities in addition to the main constituents (Mg, Si, Zr, and P) of the Sauereisen powder.

The findings discussed in the previous paragraphs show that phosphorus, a constituent of the ceramic cement, was evolved during elevated temperature exposure and was observed on the hanging wire fracture surfaces.

To determine the extent of the contamination problem, hanging wires of both varieties exposed in the A/C retorts at each of the four test temperatures were analyzed for phosphorus content. These results are shown in Table 2.4. Bulk phosphorus analysis was also carried out (Table 2.4) on the Inconel 601 wire taken from a 1050°C single specimen creep retort (ST-3) which had accumulated 300 hours of exposure. Note that the Sauereisen cement was present in each of the eight A/C retorts, but was not present in the single or multispecimen creep retorts. Inspection of these results indicates that an appreciable amount of

TABLE 2.3

SAUEREISEN #8 POWDER CERAMIC CEMENT

PHOSPHORUS ANALYSIS

<u>Retort</u>	<u>Exposure Temp. & Time</u>	<u>Wt.-% P</u>
--	As Received	4.2
A/C-6	750°C/1575 hrs.	3.63
A/C-3	850°C/1104 hrs.	2.7
A/C-2	950°C/629 hrs.	1.6
A/C-8	1050°C/585 hrs.	0.9

QUALITATIVE SPECTROGRAPHIC ANALYSIS

(AS-RECEIVED POWDER)

<u>Constituent Ranking (Limits, Wt.-%)</u>	<u>Elements</u>
Major (2 - 100%)	Mg, Si
Minor (0.2 - 20%)	Zr, P
Trace + (0.02 - 2%)	B, Fe
Trace (0.002 - 0.2%)	Ca, Al, Ti
Faint Trace (< 0.002%)	Cd, V, Co, Cr, Pd, Mn, Cu, Ni

TABLE 2.4

PHOSPHORUS ANALYSIS OF HANGING WIRES

(Bulk Analysis)

Retort	Temp. & Time	Nichrome 0.41mm (0.016 in.) Dia. Wt.-% P	Inconel 601 0.89mm (0.035 in.) Dia. Wt.-% P
--	As Received	0.003	0.011
A/C-5	750°C 1575 hrs.	0.07 ^(b) /0.14 ^(c)	0.05
A/C-4	850°C 1000 hrs. ^(a)	--	0.22 ^(d) /0.35 ^(e)
A/C-3	850°C 1104 hrs.	0.26	0.05
A/C-1	950°C 550 hrs.	--	0.08
A/C-2	950°C 629 hrs.	0.26	0.025
A/C-8	1050°C 585 hrs.	0.31	0.67
ST-3	1050°C 300 hrs.	No Nichrome Wire Present	0.011

NOTES:

- (a) Zone 4 experienced overtemperature
 (b) Wire not in direct contact with cement
 (c) Wire in direct contact with cement
 (d) Wire taken from top position of furnace
 (e) Wire taken from Zone 4 (overtemperature zone)

phosphorus was picked up by both varieties of wire after exposure in the A/C retorts at each of the four test temperatures. Additionally, although some inconsistencies exist, the results suggest increasing phosphorus content with increasing exposure temperature. Also note (Table 2.4) that no increase in phosphorus content was observed in the wire removed from the single specimen creep furnace (ST-3). This is of importance, since it indicates that phosphorus was not circulated around the loop as a constituent in the gas, but was confined to the A/C test stands.

Unetched optical photomicrographs of both types of as-received hanging wire are shown in Figure 2.1 for reference and comparison purposes. Note that except for a few fine, randomly-dispersed particles (presumably carbonitrides) evident in the Inconel 601 alloy, the structures appear essentially featureless in both cases.

Optical photomicrographs (unetched) of nichrome wire subsequent to exposure in the controlled-purity helium A/C retorts are shown in Figure 2.2. Note that morphologically similar (globular) second phase particles are revealed after exposure at each of the four test temperatures. The particles appear to be randomly dispersed throughout the wires; however, size and volume fraction of the second phase particles appear to increase with increasing exposure temperature. Microprobe analysis of the 1050°C exposed nichrome wire identified these particles as a phosphorus-nickel-chromium phase.

The results of the metallographic analysis of the Inconel 601 wire proved to be somewhat more complex than the nichrome wire findings in that carburization was apparently more severe in the former material than the latter. Optical photomicrographs (unetched) of Inconel 601 wire after exposure in controlled-purity helium at each of the four test temperatures are shown in Figure 2.3. Note that three of the wires (750°C, 850°C, and 950°C test temperatures) were exposed in A/C retorts while the wire exposed at 1050°C was removed from a single-specimen creep retort (ST-3). The microstructure of the Inconel 601 wire exposed in the 1050°C A/C retort will be discussed below, since an additional (phosphorus-containing) phase was observed in this material. Note, in Figure 2.3 the globular, intergranular (and to a lesser extent intragranular) particles which are revealed in each case. These particles are presumed to be carbides. Although the test times are varied, the degree of carburization appears more severe

Nichrome Wire

200X

Inconel 601 Wire

200X

Figure 2.1 Optical photomicrographs showing unetched microstructures of transverse cross-sections of Nichrome wire (top) and Inconel 601 wire (bottom) in as-received condition.

750°C/1575 hrs. A/C-5 1000X
(Phase contrast)

850°C/1104 hrs. A/C-3 400X

950°C/550 hrs. A/C-1 400X

1050°C/497 hrs. A/C-7 400X

Figure 2.2 Optical photomicrographs showing unetched microstructure of transverse cross-sections of Nichrome wire after exposure in controlled-purity helium A/C retorts. Exposure parameters and retort as indicated.

750°C/1575 hrs.

A/C-5

850°C/1104 hrs.

A/C-3

950°C/550 hrs.

A/C-1

1050°C/300 hrs.

ST-3

Figure 2.3 Optical photomicrographs showing unetched microstructure of transverse cross-section of Inconel 601 wire after exposure in controlled-purity helium. Exposure parameters and retort as indicated. 200X.

after exposure at the higher temperatures. The extent and morphology of the carbides observed in the Inconel 601 wire could result in poor room temperature ductility and possibly excessive creep deformation at the grain boundaries due to depletion of solid solution strengthening elements (particularly Cr) near the grain boundaries. This feature alone may limit the useful life or exposure temperature of the Inconel 601 wire. A second phase observed in this material after exposure was identified as aluminum oxide by electron microprobe. This phase appears as black, elliptically-shaped particles and can be observed predominantly around the periphery of the 950°C and 1050°C exposed wires (Figure 2.3). Additionally, a few blocky, randomly dispersed carbonitrides are also in evidence; this type of particle was observed in the as-received Inconel 601 wire.

Figure 2.4 shows an optical photomicrograph (unetched) of Inconel 601 wire after exposure at 1050°C for 497 hrs. in A/C-7. In this case, a eutectic-like phase was evident (a larger variant is shown in Figure 2.4); in addition, a number of fairly large, blocky or elongated particles were also observed. As before, aluminum oxides were revealed around the periphery of the wire (Figure 2.4) and a few nitrides or carbonitrides were also noted (not shown in Figure 2.4). Finally, a much finer phase was observed scattered throughout the structure; however, these particles were not identified because of their relatively small size.

X-ray energy spectra of the Inconel 601 wire exposed at 1050°C for 497 hrs. are shown in Figure 2.4. Note that the large blocky particles were identified as chromium carbides. Also, the eutectic-like particles were found to contain nickel, chromium and iron, as well as phosphorus.

One possible explanation concerning the somewhat different morphologies of the wire exposed 1050°C/497 hrs. as compared with those shown in Figure 2.3 may center on the degree of phosphorus pick-up (Table 2.4). Inconel 601 wire exposed at 1050°C was found to contain significantly higher levels of phosphorus relative to the wires exposed at lower temperatures. This is not surprising, since the amount of available phosphorus (evolved from the Sauereisen cement) was found to increase with increasing exposure temperature.

A number of additional chemical analyses were conducted on various alumina and metallic components. These analyses (see Table 2.5) were conducted primarily

1050°C/497 hrs.

A/C-7

- A. Optical photomicrograph showing unetched microstructure of transverse cross-section of Inconel 601 wire after exposure in controlled-purity helium. Exposure parameters and retort as indicated. 200X. Note: 1) The eutectic-like (P-rich) particle; 2) the carbides; and 3) the Al oxide particles.

Chromium carbides

Eutectic-like (P-rich) phase

Inconel 601 matrix

Aluminum oxides

Al P Cr Fe Ni

- B. X-Ray Energy spectra of Inconel 601 wire after exposure in controlled-purity helium A/C retort. Exposure parameters 1050°C/497 hrs.

Figure 2.4 Optical photomicrograph and x-ray energy spectra of Inconel 601 wire exposed for 497 hours at 1050°C in A/C-7.

to determine if there were any additional sources of phosphorus within the system. Inspection of Table 2.5 indicates that very low levels of phosphorus were found in the alumina retort components in the as-received condition. Also, low levels of phosphorus were found in the cross-pin exposed at 850°C (1562°F) for 1104 hrs., suggesting that phosphorus is not being absorbed by the alumina components. The copper turnings used in the helium purification system also revealed only baseline levels of phosphorus; and, in addition, only negligible amounts of any other "tramp" element.

Since analysis has indicated that phosphorus would deposit on the cool A/C retort exit caps, residue found on the 950°C (1742°F) and 1050°C (1922°F) top caps was analyzed for phosphorus. Additionally, a complete qualitative spectrographic analysis was also carried out on the deposit. Results are shown in Table 2.6 and indicate that a significant level of phosphorus was found in the residue removed from the top caps, suggesting that phosphorus was confined to the A/C stands. Also note that Zn was found to be a second "major" constituent of the residue. This finding was peculiar to these heat shields. The shields were all replaced in the retort cleaning procedure (Appendix) and analysis on the new shield showed that no significant Zn was present.

To determine phosphorus pick-up on actual test specimens, two approaches have been taken. The first approach involved taking thin 0.76mm (0.030 in.) slices off the ends of weldment coupons. These results, presented in Table 2.7, reveal normal or acceptable levels of phosphorus except in two cases. Abnormally high levels of phosphorus were observed on the HD-556 weldment exposed at 750°C/1000-hrs and Hastelloy X at 850°C/1000-hrs. Note that the specification maximum is 0.04 wt.-% phosphorus for both materials.

The second approach has been to analyze actual structural stability pins for phosphorus contamination. Initially, the experimental alloy, Ni-20Cr, was selected since the composition of this material was quite similar to the chemistry of the nichrome wire. The 750°C/1000-hr and 1050°C/497-hr Ni-20Cr specimens have been analyzed with results for both bulk and surface analyses shown in Table 2.8. The surface analyses were run on machined chips turned from the outer 0.76mm (0.030 in.) of the structural stability pin. Note, Table 2.8, that both the surface and the bulk phosphorus levels of the Ni-20Cr corrosion pins are fairly low. However, metallographic examination and electron microprobe analysis

TABLE 2.5

PHOSPHORUS ANALYSIS OF ALUMINA & METALLIC COMPONENTS

<u>Component</u>	<u>Exposure Temp. & Time</u>	<u>Wt.-% P</u>
Alumina Cross Pin	As-Received	<0.005
Alumina Cross Pin	850 ^o C/1104 hrs. (A/C-3)	<0.005
Alumina Vertical Post	As-Received	<0.005
Alumina Retort	As-Received	0.0025
Alumina Sleeve	As-Received	0.001
Copper Turnings	As-Received	<0.002
Fisher Copper ("Angel's Hair")	As-Received	0.009

COPPER TURNINGS: AS-RECEIVED

QUANTITATIVE SPECTROGRAPHIC ANALYSIS

<u>Element</u>	<u>Approximate Wt.-%</u>
Te	ND ^(a) < 0.03
As	ND < 0.01
Sb	ND < 0.005
Ni	0.001
Si	< 0.001
Cr	< 0.0009
Sn	0.0007
Ag	0.0006
Bi	< 0.0001
Mn	< 0.0004
Cd	ND
Pb	ND < 0.0003
<u>Wet Analysis</u>	
S Cleaned	0.003
As-Received	0.005

(a) None Detected.

TABLE 2.6

ANALYSIS OF RESIDUE FROM TOP CAPS OF
AGING/CORROSION RETORTS

QUALITATIVE SPECTROGRAPHIC ANALYSIS

<u>Constituent Ranking (Limits, Wt.-%)</u>	<u>Elements</u>
Major (2 - 100%)	Zn, P
Minor (0.2 - 20%)	Cr
Trace + (0.02 - 2%)	Pb, B, Fe, Mn, Bi, Al, Cd
Trace (0.002 - 0.2%)	Si, Ni, Na
Faint Trace (< 0.002%)	Ag, Cu, Mg, Sn

Wet Analysis for Phosphorus: ~ 7 Wt.-%

TABLE 2.7

RESULTS OF PHOSPHORUS ANALYSES ON EXPOSED
WELDMENT COUPONS ^(a)

<u>Retort</u>	<u>Exposure Temp. & Time</u>	<u>Hastelloy X</u> <u>Wt.-% P</u>	<u>HD-556</u> <u>Wt.-% P</u>
--	Maximum, Specification	0.04	0.04
--	As-Received, Vendor Certification	0.017	0.021
A/C-6	750°C/1000 hrs.	0.024	0.049
A/C-4	850°C/1000 hrs. ^(b)	0.061	0.023
A/C-2	950°C/629 hrs.	0.028	0.019
A/C-8	1050°C/585 hrs.	0.017	0.025

(a) Analysis run on 0.76mm (0.030 in.) slices machined from one end of coupons, containing the base metal only.

(b) Furnace Experienced Overttemperature

TABLE 2.8

RESULTS OF PHOSPHORUS ANALYSIS ON EXPOSED
Ni-20 Cr STRUCTURAL STABILITY SPECIMENS

<u>Retort</u>	<u>Exposure Temp. & Time</u>	<u>Wt.-% P</u>	<u>Comments</u>
A/C-6	750°C/1000 hrs.	0.002	Bulk Analysis
A/C-6	750°C/1000 hrs.	0.002	Surface 0.76mm (0.030 in.)
A/C-7	1050°C/497 hrs. (a)	0.002	Bulk Analysis
A/C-7	1050°C/497 hrs. (a)	0.012	Surface 0.76mm (0.030 in.)

(a) Furnace Experienced Overttemperature.

of the specimen exposed at 1050°C/497 hrs. revealed an atypical, localized phosphorus-rich zone near the surface of the sample. This region, which revealed both a phosphorus-containing phase and carbides, was adjacent to a surface area which appeared to have been liquated. A length of nichrome wire was also bonded to the Ni-20Cr pin in this area. Thus, phosphorus could have been transferred directly to the Ni-20Cr pin through contact with the nichrome wire. Whether or not the nichrome wire observed bonded to the Ni-20Cr pin was in immediate contact with Sauereisen cement is unknown.

Subsequent to the initial work on the experimental Ni-20Cr alloy, additional analyses were run on a number of NPH and DCHT alloy structural stability pins to try to more accurately determine the extent of contamination and, hence, the salvageability of specimens. These analyses were performed with 0.127mm (0.005-inch) surface turnings to help to identify the surface P levels. This work included surface analysis on a number of different alloys exposed at 750°C/1000 hrs. (AC-6) and 850°C/1104 hrs. (AC-3) including HD-556, Hastelloy X, Manaurite 36XS, Mo-Re 2, Rene' 80 and IN-738. These alloys were selected for analysis because phosphorus specification maxima have been set, and vendor analyses are available. Results are shown in Table 2.9 and indicate that some of the specimens have picked up phosphorus, and, in a more limited number of cases, the phosphorus content exceeded the specification limit for this element.

Based on these results, a decision was made to replace almost all of the samples already exposed in the A/C retorts. Corrosion pins and weldments exposed at 1000 hours/750°C and 1104 hours/850°C, and tensile and impact specimens exposed at 1279 hours/750°C and 1104 hours/850°C were saved for evaluation. These specimens are presently being tested and examined. All of the rest of the specimens will be replaced and the tests restarted. No test underway in the A/C retorts during contamination period will be restarted. (Note, that the tensile and impact samples which were designated for removal at 3000 hrs will now be removed at 1000 hrs throughout the rest of the screening program.) The data from the specimens evaluated after approximately 1000 hours exposure at 750°C and 850°C (1382° and 1562°F) will be reviewed with respect to the results of the new specimens (when the data are available) to assure that contamination of these specimens had not occurred and that the results were not affected by the incident. If there is evidence that the low levels of P at 750°C and 850°C (1382° and 1562°F) influenced the results, those tests will be rerun.

TABLE 2.9

PHOSPHORUS ANALYSIS ON SELECTED STRUCTURAL
STABILITY PINS

Alloy	Analysis from Structural Stability Pins As-Received and Exposed		Surface Analysis ^(a) 750°C/ 1000 hrs. A/C-6		Surface Analysis ^(a) 850°C/ 1104 hrs. A/C-3
	Maximum Specification Wt.-% P	As-Received Bulk Analysis Wt.-% P	Wt.-% P		Wt.-% P
		Vendor	GE		
Ni - 20 Cr	--	--	--	0.002 ^(b)	0.019
HD-556	0.04 Max.	0.021	0.020	0.04-0.032	0.034
Hastelloy X	0.04 Max.	0.017	0.015	0.05-0.026	0.034
Manaurite 36XS	0.03 Max.	0.026	0.016	0.020	0.018
Mo-Re 2	0.04 Max.	0.015	0.032	0.040	0.029
Rene' 80	0.015 Max.	<0.01	0.006	0.079	0.114
IN-738	0.015 Max.	0.006	0.006	0.030	0.031

(a) Taken from 0.127mm (0.005 in.) surface turnings.

(b) Analysis run on 0.76mm (0.030 in.) surface layer.

NOTE: P analysis capability was ± 0.002 wt. % P
for analysis performed at GE.

Since the A/C retort components also were exposed to phosphorus, it was necessary to thoroughly clean or replace the retort components. The retorts, hanging rods and crosspins are ceramic (α -alumina), while the heat (radiation) shields, retort end caps, gas supply and return lines, and the sheathed thermocouples are metallic. All replaceable components, including the ceramic crosspins, the heat shields and their spacers, the outlet gas line and the thermocouples, were replaced. The other components were washed and rinsed several times with distilled water to remove any traces of adsorbed phosphorus or P_2O_5 which are soluble in water at room temperature.

To verify the cleanliness of the retort systems after cleaning, part replacement and reassembly, 0.41 mm dia. (0.016 inch dia.) Nichrome wire is exposed for at least 100 hours at 1050°C (1922°F) in flowing purge gas (He with approximately 500 ppm H_2 and < 3.0 ppm H_2O). After the exposure, the wire is analyzed for phosphorus. A retort is certified to be clean if there is no detectible increase of phosphorus in the wire's phosphorus content over the as-received value. The limit of phosphorus detectability is $\pm 0.002\%$, and the as-received phosphorus level in the Nichrome wire is 0.003%.

The entire retort cleaning procedure is included as an appendix to this report.

At the end of the quarter all retorts were in the process of being cleaned.

Aside from phosphorus contamination, an additional problem unearthed by metallographic analysis during the course of this investigation was the carburization of the hanging wires. Analyses for carbon on both varieties of hanging wire after exposure at each of the four test temperatures are shown in Table 2.10. Inspection of these results clearly indicates that, at least at the highest test temperature, a significant amount of carbon is being picked up by the hanging wires. Additionally, carburization seems to be more severe for the Inconel 601 wire than the nichrome material. Since the carburization problem alone may limit the useful life of the wires, a search for a new hanging wire has been initiated. Creep tests will be run in the helium loop atmosphere on a variety of wires (Inconel 601, platinum - 13% rhodium, nichrome, nickel, molybdenum-rhenium, Rene' 41, chromel, and alumel) to identify a suitable hanging wire material.

TABLE 2.10

CARBON CONTENT OF HANGING WIRES

<u>Retort</u>	<u>Temp. & Time</u>	<u>Nichrome Wt.-% C</u>	<u>Inconel 601 Wt.-% C</u>
--	As Received	0.006	0.028
A/C-5	750°C/1575 hrs.	0.090 to 0.312	0.131
A/C-3	850°C/1104 hrs.	0.067	0.319
A/C-2	950°C/629 hrs.	0.022 to 0.077	0.368
A/C-7	1050°C/497 hrs.	--	0.498 to 0.529
A/C-8	1050°C/585 hrs.	0.304	--

2.7 TASK 9 - DATA MANAGEMENT AND PROGRAM MANAGEMENT

2.7.1 Data Management

Work on the data base definition file continued during the quarter.

2.7.2 Program Management

The following program meetings were conducted or attended by program staff during the reporting period:

- HTGR Materials Design Data Needs Meeting was attended at General Atomic on July 18, 1979. Design requirements for the HTGR-Gas Turbine were reviewed by General Atomic staff followed by discussions on recommendations for materials data needs. Representatives from DOE, GCRA, GAC, ORNL and GE were in attendance.
- A program review meeting was held at General Electric in Schenectady, NY with J.E. Fox, DOE Program Manager on September 5 and 6, 1979.
- HTGR Base Technology Meeting was attended at Oak Ridge National Laboratory on September 12 and 13, 1979. Discussions included impurity analysis procedures in HTR helium and recommendations for materials to be selected for the General Electric Phase 2 test program.
- A meeting was held with Climax Molybdenum at Ann Arbor, Michigan on September 20, 1979. Advances in coating technology for TZM alloys were reviewed as well as discussions on new high temperature alloys.

In addition to the meetings listed above, weekly meetings were held with the General Electric program personnel in Schenectady, NY to review technical results and program status.

Prime responsibility for the DCHT Materials Investigation has been transferred from the General Electric Aircraft Engine Business Group, Cincinnati, Ohio to the Gas Reactor Materials Unit, Energy Systems Programs Department, Schenectady, NY. Also, since all program activities will now be centered in General Electric laboratories in Schenectady, NY, steps were taken to transfer the Data Management task from the Aircraft Engine Business Group in Cincinnati, Ohio to Schenectady, NY. Key contributors to the program from the Aircraft Engine Business Group will continue to provide technical input to the program on a consulting basis.

On August 29, Amendment A011 to the Contract was negotiated to provide funds for needed items of capital equipment. These items are:

1. Stereo Macroscope
2. Ten (10) Extensometers for Creep Retorts
3. Gas Chemistry Data Handling System
4. Helium Supply and Analysis System for Fatigue Retorts
5. Two (2) Portable Gas Analysis Systems
6. Moisture Analysis Calibration System

The Program Master Schedule is presented in Figure 2.5. This schedule reflects the changes that were required due to the shutdown of the test loop in the May-June 1979 time period, and the need to restart the majority of the structural stability (A/C) tests.

Section 3.0

WORK TO BE COMPLETED DURING THE NEXT QUARTER

3.0 WORK TO BE COMPLETED DURING THE NEXT QUARTER

- Structural stability specimens, tensile specimens and impact specimens exposed for 1000 hours at 750°C, 1279 hours at 750°C and 1104 hours at 850°C will be examined and tested.
- Selected aging/corrosion tests will be restarted and sufficient specimens for the restart of the remaining tests will be machined,
- Two 3000-hour TZM screening creep tests will be completed.
- The remaining two multispecimen creep stands, at 950°C and 1050°C, will be started.
- Alloys for the intensive screening testing phase will be selected.
- Modifications to Loop No. 2 will continue.
- The design of a once-through-then-out He supply for the fatigue test stand will be finalized.

Section 4.0

APPENDIX

**AGING/CORROSION RETORT
AND
TEST STAND CLEANING PROCEDURE**

CLEANING PROCEDURE FOR AGING/CORROSION RETORTS

1. Remove ceramic specimen support rod and store test specimens.
2. Disassemble specimen support. Remove and discard alumina crosspins, minimizing contact between residual Sauereisen cement and ceramic centerpost which will be re-used. Thermocouples should be removed with a minimum of bending (none, if possible), tagged with stand and thermocouple numbers, and set aside. Radiation shields should be removed along with spacers and support pins. These parts should be tagged as to which retort they came from and whether they are upper or lower shields. The center support rod should be disconnected from the universal joint at the upper end cap.
3. Remove lower end caps. Remove and preserve o-rings.
4. Disassemble upper end caps by removing thermocouple (TC) ports. Viton TC seals should be discarded.
5. Clean top and bottom end caps as follows:
 - a. Soak overnight in distilled water, then scrub with distilled water and a stiff, non-metallic bristled brush. Repeat until there is no visible residue.
 - b. Rinse, then soak overnight in distilled water, drain and air dry with a heat gun.
6. Disconnect helium lines. The helium inlet line should be disconnected at the lower retort end fitting. Cap the line and install a temporary plug in the end fitting. The helium exhaust line should be disconnected at both the retort and utility frame sub-assembly ends and will be discarded. Install a temporary plug in the retort end fitting and cap the utility frame sub-assembly port.
7. Install the retort lower end cap.

8. Place the specimen support rod in the retort. Lower the tube gently into the retort using a loop of Inconel 601 wire through the top holes of the ceramic tube until it rests on the lower end cap and allow it to lean against the retort wall.
9. Fill the retort to its upper lip with distilled water and soak overnight.
10. Drain the distilled water, remove the ceramic specimen support rod, and scrub all accessible surfaces with a stiff, non-metallic bristled brush. Rinse with fresh distilled water.
11. Replace the specimen support rod in the retort as in Step 8, then repeat Step 9.
12. Drain the distilled water, remove the specimen support rod, and lower end cap, then rinse all wetted surfaces with fresh distilled water from a squirt bottle. Allow components to air dry overnight.
13. Replace radiation shields with new shields.
14. Reassemble the test retort. Use new thermocouples; clean and regrease O-rings. Install new ceramic crosspins.
15. Remove the temporary helium port plugs. This will allow circulation of air through the retort.
16. Apply heat. Manually set power levels to all five zones. Begin with 1%, monitor the furnace thermocouples, and adjust power as required until all TC's indicate $66^{\circ}\text{--}93^{\circ}\text{C}$ ($150^{\circ}\text{--}200^{\circ}\text{F}$). It is important not to exceed 100°C (212°F) at this stage of dry-out. Maintain $66^{\circ}\text{--}93^{\circ}\text{C}$ ($150^{\circ}\text{--}200^{\circ}\text{F}$) at least 24 hours.
17. Apply supplemental heating to the end caps by judicious use of a hot air gun. It will be necessary to shut off cooling water flow for this step. Allow each end cap to attain approximately 93°C (200°F) as measured by a thermocouple probe held against its outer surface. Do not exceed 93°C (200°F) as this might endanger the O-ring seals. Maintain the end cap temperature in the $66^{\circ}\text{--}93^{\circ}\text{C}$ ($150^{\circ}\text{--}200^{\circ}\text{F}$) range for at least four hours. This may be accomplished near the end of the 24 hours specified for Step 16.

18. Replace the temporary helium port plugs. Evacuate the retort and hold under vacuum at about 93°C (200°F) for at least ten hours.
19. Remove cap from helium inlet tube and reconnect to lower end cap fitting.
20. Fabricate a new helium exhaust line and install.
21. Connect the portable gas analysis station (GAS) to the retort exhaust valve and a vacuum pump to the retort vacuum pump.
22. Evacuate and refill the retort three times with purge gas, then establish approximately 250 cc/min of purge gas flow through retort and GAS. If the preliminary DPM reading indicates more than 200 ppm of H_2O in the retort effluent, do not leave that instrument exposed to the gas stream.
23. Alternate evacuation, refilling with purge gas, and monitoring retort effluent until the DPM indicates H_2O levels are below 100 ppm. Maintain retort temperature at $66^{\circ}\text{--}93^{\circ}\text{C}$ ($150^{\circ}\text{--}200^{\circ}\text{F}$).
24. Increase retort temperature to 204°C (400°F) (manual or automatic control as desired) and monitor H_2O output. Maintain temperature until H_2O is less than 50 ppm.
25. Ramp temperature to 1050°C (1922°F) at $28^{\circ}\text{C}/\text{hour}$ ($50^{\circ}\text{F}/\text{hour}$) (maximum). Be prepared to interrupt ramp and hold at any temperature where H_2O level increases to over 50 ppm. Hold at 1050°C (1922°F) for 24 hours, then cool slowly to room temperature.

This completes the cleaning procedure. A test designed to verify the adequacy of the cleaning procedure is to be conducted as follows:

1. Prepare several (5-10) test specimens. Each sample consists of a 12 cm (5-inch) length of 0.406 mm (.016 inch) diameter Nichrome V wire. Samples should be subjected to the regular specimen cleaning procedure.
2. Test specimens should be installed on the top crosspin of the specimen support. They may be bent to any convenient shape, but it is desired to have them intersect as much of the gas flow path as possible. Thus, a completely vertical specimen orientation is least desirable and a wholly horizontal position would be most effective.

3. Assemble and leak check the retort and follow normal test procedures to expose the samples to at least 100 hours at 1050^oC (1922^oF) with 100 cc/min of purge gas.
4. Retrieve specimens after exposure and conduct chemical analysis for P.
5. If there is no measurable increase in the P level of the wire, the retort is certified to be clean and ready for further testing.