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GAS-COOLED REACTOR PROJECT
QUARTERLY PROGRESS REPORT
FOR PERIOD ENDING JUNE 30, 1960

OAK RIDGE NATIONAL LABORATORY

operated by

UNION CARBIDE CORPORATION

for the

U.S. ATOMIC ENERGY COMMISSION

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Staff
Oak Ridge National Laboratory

DATE ISSUED

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SUMMARY

Part 1. Design Investigations

1. Reactor Physics

The effects on the power distribution of operating the EGCR without experimental fuel assemblies installed in all eight in-pile loops were investigated. It was found that the 5-in.-diam loops near the center of the core have a peaking effect on the radial power distribution when loaded with experimental assemblies, whereas the 9-in.-diam loops have a flattening effect. When the loops are empty, their effects are the reverse because of the poisoning of the reactor by the permanent, steel through-tubes.

The effects on the EGCR power distribution of inserting control rods in various patterns have been studied. In order to represent the probable startup conditions, it was assumed that the four large through-tubes were filled with graphite and that the four small through-tubes contained homogeneous graphite-U²³⁵ assemblies with a carbon-to-uranium ratio of 200 (0.3 kg of U²³⁵ per foot). The selected control configuration satisfies the condition that the criticality of the reactor be independent of the continued future operation of the loop experiments.

The nonuniform neutron absorption on the surface of EGCR control rods was investigated. Peaking factors have been obtained that describe the variation of the neutron absorption rate at the tip of a partially inserted EGCR control rod and azimuthally around fully inserted rods at several locations within the core.

Neutron spectrum calculations have been carried out for several moderator temperatures and absorption cross sections in order to determine for graphite-moderated systems the adequacy of the usual approximation of the spectrum as the sum of a Maxwellian distribution and a 1/E distribution terminated at the low energy end by a suitable joining function (or functions). A preliminary analysis has indicated that a single joining function can be found that will adequately represent the calculated

spectra. It was assumed in these calculations that the moderator was an infinite carbon-gas medium.

A series of calculations has been started to explore the nuclear characteristics of two-region, semihomogeneous, gas-cooled, BeO-moderated reactors in which the fissionable material ($U^{233}O_2$) is in a central cylindrical core and the fertile material (ThO_2) is entirely in a surrounding blanket. Such complete segregation of the fissionable and fertile material has been found to result in a high conversion ratio but a low thermal power output. Multiple-region reactors will be examined as a means of increasing the power output while retaining the high conversion ratio.

2. Reactor Design Studies

The Title II design of the EGCR fuel assembly has been completed. The shear pins and bending tabs provided to assure that it will be possible to remove a "jammed" assembly from a fuel channel were designed on the basis of data obtained in load tests. Pressure drops have been determined for the new design, and tests of coolant flow distribution in the fuel rod cluster have been initiated. Preliminary results indicate that there will be no severe maldistributions of flow for 0-, 30-, 60-, and 90-deg relative orientations between assemblies.

An analysis has been made of the nominal maximum fuel element surface temperature as a function of the channel mixed-mean gas temperature and the maximum channel exit gas temperature for one mode of control of EGCR operation. It was assumed that the central control rod was fully inserted, the four control rods in the inner ring were one-half inserted, and the remaining control rods were in the upper reflector. The temperature control that could be achieved by variations in channel orificing was studied.

Heat transfer data for rod clusters have been analyzed, and a correlation of the average heat transfer coefficient with Reynolds number and axial position has been established. A study of radiative heat transfer among the seven rods and sleeve of the EGCR fuel cluster has

resulted in view factors for the various surfaces that take into account emissivities, geometrical configurations, and other factors and are independent of temperature. An estimate of the effect of radiation on the diametral temperature difference across an outer rod of the cluster indicates that the temperature difference based on flux peaking in the outer rods may be diminished by about 20°F by radiative heat transfer.

A mathematical model has been developed for studying shifting of the coolant stream as it moves along a rod in order to predict the temperatures of the parallel streams as they progress through the reactor. Temperatures are being calculated for various rod spacings, hanger and spacer effects, flux distributions, and gas flow rates, as well as for variations in other parameters.

A fuel element life code developed for computing the internal temperature structure, the amount of fission gas released, the internal pressure, the cladding strain when the internal pressure exceeds the coolant pressure, and the creep damage was used for comparing top-loading and inverted-loading fuel programs for the EGCR. The inverted-loading program was shown to be the more desirable in that it results in lower fuel element surface temperatures and permits the use of UO_2 with an effective fission-gas diffusion coefficient a hundredfold larger than that required for the top-loading program.

A statistical method has been developed for estimating the probability that the hot spot on the EGCR fuel element will exceed a given temperature. Probabilities have been calculated on the basis of the uncertainties in the ten independent variables on which the fuel element surface temperature depends. This analytical method has the advantage that it does not assume that all factors attain their worst values at the same spot and time.

A method of cooling the EGCR control rods has been developed that will minimize diversion of coolant flow through leakage paths between graphite blocks. This method consists of directing a portion of the coolant flow for the control rod drive mechanism down the shroud and through the inside of the hollow control rod. This flow would leave the

bottom of the rod and flow up the outside of the rod to the top plenum. A preliminary design of a control rod cooled by this method has been developed.

Means for reducing the thermal stresses in the top head nozzles of the EGCR pressure vessel were studied. It appears that the thermal gradients could be reduced (and therefore the stresses) by (1) relocating the insulation in the nozzles to insulate them from the incoming coolant flow and (2) increasing coolant flow to the nozzles near the center of the vessel and reducing the flow to those at larger radii.

The stresses in the graphite sleeves of the EGCR fuel elements were calculated, and the maximum stress was found to be within the allowable limit. The structural behavior of the long (19 ft 4 in.) graphite columns to be used in the EGCR core was also considered. A test conducted on a 1/16-scale model demonstrated that the present design will fulfill the structural requirements.

A study was made of the thermal stresses in the EGCR pressure vessel support skirt, and a satisfactory design was developed. It was found to be impractical to provide enough insulation on the top segment to reduce the stresses to acceptable levels, so the conductance was increased by adding an overlay of pure copper in the region of the joint between the vessel and the skirt.

Procedures for removing ruthenium and cerium contamination from steel have been outlined and incorporated in procedures for decontaminating the EGCR charge and service machines. The same general methods of decontamination can be used for the in-pile loops. Further the loops could be filled with strong acid solutions, if necessary.

3. Experimental Investigations of Heat Transfer and Fluid Flow

Experimental information has been obtained on the thermal characteristics of the specified EGCR fuel cluster. Circumferential temperature profiles have been plotted as a function of axial position for two peripheral tubes of the cluster spaced at an inner-to-outer ligament ratio of 2 and containing a 60-deg-pad spacer. The Reynolds modulus (50 000) and

surface thermal flux ($6500 \text{ Btu/hr}\cdot\text{ft}^2$) were found to be consistent with the values proposed for the EGCR. An 8 to 14°F difference between the mean temperatures of the two tubes has been ascribed to a residual entrance maldistribution in the flow. This condition may also be reflected in a progressive (with axial location) counterclockwise rotation of the profiles for tube 6 and a similar clockwise rotation for tube 4. The 60-deg-pad spacer results in a complete upset in the surface temperature profiles downstream of the spacer as compared with the data obtained previously with an adjustable, minimum-obstruction spacer. The temperature minimum now appearing at the 180-deg position may be related to the increased turbulence in the wake of the spacer pad. A marked flattening of the temperature profiles as the flow proceeds downstream has again been observed.

The effect of relative orientation of adjacent clusters on the heat-transfer distribution in the downstream element has been studied by means of mass-removal measurements on naphthalene-coated rods. End spiders of EGCR Title II design with conical bosses were used along with the 60-deg-pad mid-cluster spacer. Initial results at a distance of $3.8 L/d_e$ from the bundle entrance with a 0-deg rotation exhibit a trifoliate pattern for all rods, with the maxima corresponding to the three regions of relatively unobstructed flow around each rod. A comparison of the circumferential-mean mass-transfer factor with data on entrance-region heat transfer in circular tubes shows striking agreement. Further comparison with the experimental heat-transfer profiles indicates general similarity; explanation of the differences observed awaits assessment of the relative roles of fluid turbulence in the wakes of the spider ribs in increasing mass transfer and of wall conduction in decreasing heat transfer at the same circumferential position.

Velocity distributions in the downstream element of two adjacent EGCR-type clusters have been determined with Title I H-bar, Title II cylindrical, and Title II conical spiders at various relative orientations. The flow field was found to be much more complex than had

previously been observed under "idealized" conditions with minimum-resistance spiders. Results with the H-bar spider showed a serious flow deficiency in the interior flow zone that could cause excessive temperatures on the central tube and along the inner side of the peripheral tubes; the minimum velocity in this region occurred for a 60-deg relative spider displacement. The velocity in the inner region increased by 25% for the Title II spider at the same orientation; the maximum interior velocity with this spider existed at a 90-deg displacement. A continuous redistribution of the flow was observed with the mean flow in the vicinity of the central tube increasing with distance from the cluster inlet. Filament visualization studies verified the existence of radial and rotational components in the flow. Maximum transverse flow with the Title I spider was found for a 15-deg spider rotation.

Part 2. Materials Research and Testing

4. Metallurgical Investigations

The recycling of scrap produced during the fabrication of UO_2 pellets for fuel elements is being studied. Attempts are being made to produce UO_2 powder of the same quality as that obtained from the initial conversion step.

The expansion of UO_2 pellets with "dished ends" has been measured and found to be about 10% less than that of standard flat-end pellets at high temperatures. Simulated capsules were thermally cycled to induce cracking of the UO_2 , and the amount of fine fragments that resulted was found to be insignificant. A precollapsed capsule and a capsule pressurized externally at 300 psi were also tested in thermal cycling. The expansion was determined to be dependent on the cladding temperature, as well as on the temperature of the UO_2 .

Studies of fission-product release upon heating of UO_2 specimens previously irradiated at low temperatures have been continued. A characteristic burst of activity is always detected during initial heating, and subsequently the activity is released at a rate that is proportional

to the square root of time. The D' values calculated from the slope of the linear portion of the curve can be correlated with temperature. The rate of fission-gas release was found to increase drastically when the sintering temperature of the UO_2 was exceeded.

Efforts to develop high-density, low-porosity graphite have continued, and graphite bodies with fired densities as high as 2.0 g/cm^3 have been formed. Various binder materials and methods of mixing are being tested.

The reaction of UO_2 with graphite has been studied over a range of temperatures, particle sizes, and pressures. A correlation of the data has permitted the evaluation of a temperature-dependent rate constant for the reaction.

In studies of the mechanical properties of graphite, tensile tests have been run at room temperature and at 750°F , and incremental loading tests have been performed at 78, 750, and 1100°F . The results of these tests indicate that the fracture stresses and strains of graphite are not temperature dependent up to 1100°F and are not time dependent, at least over the three orders of magnitude difference between the periods of the two types of tests.

The conditions required for shape-casting of small-diameter rods of UC in copper molds have been determined. Several shapes have been cast, and chemical analyses and metallographic studies are under way.

Fabrication of pure BeO and fueled-BeO specimens by extrusion and isostatic pressing and by compacting in a die are being investigated. The most suitable method seems to be compacting with a binder and sintering. The fuel is preferably added as UO_2 .

The creep testing of type 304 stainless steel has continued, and the results have further substantiated the previous data. In the study of the reactions of type 304 stainless steel with CO- CO_2 mixtures, the exposure of thin-walled type 304 stainless steel to a mixture of CO and CO_2 showed that carburization depends on the CO-to- CO_2 ratio and that it may be controlled. In-pile and out-of-pile tube-burst tests are continuing in an effort to explain the variations in strength ob-

tained for chemically similar specimens of different lengths from different heats. The effects of prior history and test environment are also being investigated further.

The effects of oxygen, nitrogen, and hydrogen on the mechanical properties of columbium are being determined in bend tests and tensile tests at room temperature and at high temperatures. Oxygen additions of up to 3000 ppm have been found to strengthen columbium without seriously affecting the ductility. Nitrogen also increases the strength, but amounts in excess of 1000 ppm seriously reduce the ductility. A significant quantity of hydrogen is absorbed by columbium in the temperature range of 500 to 600°C, and amounts above 500 ppm seriously embrittle and weaken the material.

The nil-ductility transition temperature of as-received type SA-212, grade B, steel plate was determined in drop-weight tests on 4-in.-thick specimens to be 10°F at the surface and 30°F at the midsection. A total of 150 Charpy V-notch specimens was prepared for radiation-damage studies of heat-affected zones.

The fabrication and inspection of 50 prototype EGCR Title I fuel elements were completed. Of the total of 350 capsule closure welds inspected in this work, only 6 were defective. They were rejected by the leak test inspection. Inspection and evaluation of Title I graphite fuel element sleeves supplied by three vendors revealed many cracks in sleeves from one vendor and low-density inclusions in sleeves from the other two vendors. Eddy-current inspections gave poor correlation with the size of the low-density inclusions.

A remote welding technique is being developed for welding of the EGCR experimental loops. An edge-weld made with a conventional welding torch that has a pull-type wire feed is being tested. Suitable conditions for producing linear joints have been developed, and specimens for concentric-tube welding experiments are being prepared.

A full-size mockup of one quadrant of a burst-slug-detection tube nozzle and a mockup of the thermocouple penetrations were fabricated to

confirm the procedures developed previously. A procedure was developed for the joining of type SA-212, grade B, steel to type 304 stainless steel in the 1G position using BP-85 electrode material. Procedures for other positions and for joining other steels are being developed.

A process is being developed for closure welding of fuel capsules at reduced pressures. Capsules have been welded with helium pressures ranging from 250 down to 1×10^{-3} mm Hg.

The oxidation of beryllium in CO_2 at a pressure of 1 μ was investigated, and oxidation-rate equations were formulated. In tests of the compatibility of beryllium with stainless steel in a NaK environment, the transfer of beryllium to form NiBe on the surface of the stainless steel was very slight at 600°C, but a 0.010-in. layer of NiBe was formed at 800°C.

Beryllium tubing purchased from several vendors is being evaluated by radiographic, ultrasonic, dye-penetrant, and eddy-current techniques. Up to 33% of some batches has been rejected for excessive variations in wall thickness.

Several brazing alloys suitable for joining beryllium have been developed; however, the alloys are brittle and subject to cracking. Examination of a series of diffusion-bonded specimens confirmed that good bonds may be made by heating in helium or vacuum. A tube-burst specimen of beryllium tested at 750°C failed in 3.2 hr in the tube wall without affecting the diffusion-bonded end closure.

The fusion welding of beryllium using an edge-weld closure design is currently favored for use on capsules for irradiation tests. Leaktight joints have been made on all tubing received recently. Porosity and root cracking are pronounced in welds on extruded tubing. Leaktight joints have also been made by upset welding, using a deformation pressure of 3000 psi at 550°C; however, the joint produced is mechanical rather than metallurgical.

5. In-Pile Testing of Components and Materials

Irradiation of eight full-diameter EGCR prototype group II fuel capsules has continued, and eight group III capsules are being prepared

for irradiation. Six of the group III capsules will consist of UO_2 pellets clad with beryllium. The remaining two capsules will consist of stainless-steel-clad UO_2 pellets of different grain sizes. Four capsules were inserted in the ETR during the quarter, and three were relocated to higher neutron-flux positions.

In the miniature capsule fission-gas-release experiments, irradiation of assembly L-25ab was completed and irradiation of assembly L-28ab was started. Measurements of the central fuel temperature during irradiation of assembly L-25ab indicated that the thermal conductivity of the UO_2 did not change. The tungsten-rhenium thermocouple in the a capsule gave reasonably constant readings throughout the irradiation period and was in good condition after removal from the reactor. On the other hand, the thermocouple in the b capsule failed during operation. Examination after removal from the reactor showed that the entire tantalum sheath in the heated zone and part of the rhenium wire had disintegrated.

A second fueled-graphite assembly is being prepared for irradiation in the MTR.

Measurements have been made of the fission gas evolved by miniature capsules irradiated at maximum central UO_2 temperatures in the range 2750 to 3300°F. The data indicate that, at 2900°F and above, fission-gas release increases substantially. Fission-gas samples have been obtained from ORR-irradiated capsules O-1, -2, -3, and -5 and from ETR-irradiated capsule E-1. Gamma-ray spectrometer analyses have been completed and the samples are being processed for mass spectrometer analyses. The E-1 capsule, which contained solid pellets and had very little free volume, had an internal gas pressure at room temperature of approximately 61.5 psia.

Examinations of the fueled-graphite experimental assembly (ORNL-MTR-1) that was irradiated in the MTR have been made. Visual inspection showed no changes in the graphite capsules and the carbon spacers between them. The graphite- UC_2 fuel cylinder has been removed intact from one capsule, and it shows no apparent damage. Micrometer measurements have indicated a slight decrease in diameter and a slight increase in length.

Two UO_2 samples encased in siliconized silicon carbide-coated graphite are being irradiated in the ORR closed-cycle loop. After irradiation for more than 400 hr at 1800°F there has been no increase in fission-product activity of the coolant.

A study of the release of iodine from the high-density UO_2 being irradiated in the ORR instantaneous fission-gas release experiment has indicated that iodine release has the same temperature dependence as that of the release of xenon and krypton. A massive burst of activity that occurred when the fuel temperature was increased 400°F demonstrated an abrupt change in the fission-gas retention characteristics of the fuel. Further, a temperature excursion was observed during a recent insertion of the fuel into the reactor that could only be explained as a release of stored energy. The release of stored energy indicates that the oxygen-to-uranium ratio of the fuel (initially 2.01) has increased to 2.08 or more. It appears therefore that the increased fission-gas release and the large burst of activity are the results of oxidation rather than radiation damage. The sweep gases are purified, but even 1 ppm O_2 in the gas stream could appreciably change the oxygen-to-uranium ratio over a period of months.

In-pile tube-burst-type stress-rupture tests are under way on type 304 stainless steel tubing of the type to be used as cladding material for EGCR fuel elements. Specimens are being prepared for out-of-pile tests under similar conditions.

Heavy steel plate weldments are being prepared for irradiation tests. Long-term exposures are planned in order to obtain data for extrapolation to engineering design for 20 years of service at low neutron fluxes.

The first high-temperature beryllium irradiation experiment was terminated after a fast-neutron exposure of 7×10^{19} neutrons/cm² rather than the planned exposure of 10^{21} neutrons/cm² because of the failure of furnace circuits. The apparatus is being modified for a second experiment. Density measurements on the irradiated specimens have shown

that swelling of the beryllium coupons occurred. The average density decrease was less for specimens machined from hot-pressed block than for wrought specimens.

In-pile tests of Inconel specimens continued. Specimens are being fabricated from special boron-controlled heats for a new series of in-pile tests. The out-of-pile tests of specimens from these special heats have been started.

6. Out-of-Pile Testing of Materials and Components

Compatibility tests of graphite, structural materials, and helium have demonstrated that in a reactor system such as the EGCR the area of metal surface exposed at temperatures approximating the exit gas temperature will have an important bearing on the corrosion behavior of the hotter fuel cladding. In polythermal pot tests in static helium, increasing the amount of metal held at 1100°F served to gather oxygen-containing gas contaminants at a faster rate and thereby reduce the oxidation of the hotter surfaces of the simulated fuel element held at temperatures in the range 1500 to 1800°F. The carburization of the heated element was faster in the absence of an oxide film.

Low-pressure thermal-convection loop tests with helium containing controlled amounts of impurities are continuing. The effects of pure CO and of H₂O on plain carbon and low-chromium-content steels are being studied.

Tests of two high-pressure thermal-convection loops containing helium, AGOT-grade graphite, metal test specimens, and a dummy fuel element assembly have been completed. After 1000 hr at a hot-leg temperature of 1400°F in one test and 1100°F in the other, the stainless steel specimens were covered with tightly adherent, iridescent surface films. The low-alloy steels had loosely adherent, silvery gray films that spalled when the samples were removed from the loop. The dummy element tested at 1400°F contained four rods loaded with UO₂ pellets, and it was found that the rods had collapsed around the pellets and that

bowing had occurred. No carbon mass-transfer deposits were found in the cold legs of the loops.

A small-scale gas-purification system of the type proposed for use with the EGCR coolant system has been installed on the forced-circulation loop. Preliminary tests for determining optimum operating conditions have been initiated.

In the experimental studies of the degassing behavior of graphite, several specimens prepared from needle coke, a highly purified AGOT-grade sample, a low-permeability specimen, and a coated specimen were tested. It was found that all graphites prepared from needle coke did not have the same degassing characteristics. Comparisons of the volumes of gas evolved through 1000°C by external resistance and induction heating have consistently shown that induction heating results in the evolution of at least twice as much gas as does external resistance heating at a common temperature. Data for the individual samples are presented, as well as plots giving the relationship of the total volume of gas evolved to the time of heating.

Experimental studies of the interdiffusion of argon and helium through an AGOT reactor-grade graphite septum under uniform total pressure conditions have continued, and an unexpected drift of molecules has been observed. The drift is such that all ratios of the apparent coefficients for helium to those for argon at equivalent conditions of temperature and pressure are approximately equal to the square root of the ratio of the molecular weight of argon to that of helium. At this stage of the investigation it can be concluded that the total transport of either gas depends (1) on a net drift that can be varied by imposing small pressure drops across the septum but which is not zero under uniform pressure conditions and (2) on a classical diffusion term which differs from handbook values only through the internal geometry of the graphite.

The mechanisms involved in the release of fission products from unclad high-temperature fuel elements in a gas-cooled reactor are being

studied. Indications of significant changes in the mass-transport mechanisms as a function of temperature have been obtained.

The chemical behavior of fission products in fissioning UO_2 is being studied. The rare earth oxides will "grow in" during fissioning, but they will not melt at the anticipated EGCR operating temperatures. Fission-product molybdenum, cesium, rubidium, technetium, ruthenium, rhodium, and palladium will appear as metals, with a combined yield of approximately 65 atoms per 100 fissions. It is possible that these metals will form a liquid alloy, and it seems likely that the alloy will be largely retained by the porous fuel matrix.

Methods of removing fission-product gases from the coolants of gas-cooled reactors are being investigated. A generalized Oracle program for adsorber evaluation was prepared.

In analytical chemistry studies, a thermal-conductivity apparatus has been developed for chemical analyses of mixtures of helium and argon. Techniques have been developed for preparing synthetic gas mixtures for calibration of instruments for maintaining atmospheres of controlled composition. Further, a sequential plan for sampling and testing batches of UO_2 pellets for potential use in the EGCR has been developed that minimizes the analyses required and insures that pellets are rejected if the oxygen-to-uranium ratio exceeds 2.02.

Studies of the factors that influence the accuracy of thermocouples attached to metal surfaces in high-velocity gas streams were continued. High-temperature furnaces for testing thermocouples for use in the temperature range 1000 to 3000°C are being developed. Helical tungsten-rod heating elements mechanically attached to water-cooled copper blocks have operated at temperatures above 2800°C for several days without failure. Additional tests have been made of Chromel-P-Alumel thermocouples in stagnant helium at 1000°C. It has been found that contaminants affect, but are not responsible, for the observed drift in the temperature readings.

7. Development of Test Loops and Components

Installation of gas-cooled in-pile loop No. 1 and its auxiliary equipment at the ORR is nearing completion. Results of thermocouple calibration tests have indicated that fuel element cladding temperature measurements accurate to $\pm 20^{\circ}\text{F}$ can be obtained. Design of the primary components of loop No. 2 has been completed, and equipment fabrication is under way.

Developmental work leading to the design of the EGCR experiment loops continued. The temperature profile along the length of a small through-tube was determined in order to obtain the heat-loss data needed for designing the bellows required for accommodating thermal expansion. The total compressor head required for a loop was determined on the basis of the head losses and pressure drops for individual components with hydrogen, helium, and CO_2 as the coolants.

Requirements for a hydrogen-cooled loop were developed according to the basic criteria used for the design of the helium- and CO_2 -cooled loops. It was found that the 2% criterion for pumping power should be reduced to 1% for the hydrogen-cooled loop. Three compressors in series will be required.

An analog model of the loops is being developed for studying control schemes. It appears that the responses of the loops will be sluggish because of the long lengths of heavy-walled pipe and the relatively long gas-transit time.

Design work continued on gas filters, the gas heater, loop valves, and the leak detection system. The supporting facility designs being prepared by Kaiser Engineers were reviewed. The design of the reactor service machine has been modified to provide for servicing the through-tubes.

Design studies were continued on the coolant cleanup system for the loops. Copper oxide is being evaluated as an oxidizing agent for low concentrations of H_2 and CO in helium. A fission-gas collection system has been sized for effective holdup of the fission products released from experimental fuel assemblies. Service testing of mechanical joints for loop application has continued.

Tests of compressors for ORR in-pile loop No. 1 were continued, and difficulties were encountered because of inadequate coolant flow. Methods for solving the problem are being investigated. Fabrication of the facility for testing the Bristol-Siddeley gas-bearing compressor was completed. Negotiations were initiated for procurement of the compressors for ORR in-pile loop No. 2 and the EGCR experiment loops.

Additional data were obtained on back-diffusion through labyrinth seals. Various differential pressures are being used in these tests in order to establish the limits at which back-diffusion occurs.

Part 1. Design Investigations

1. REACTOR PHYSICS

EGCR Power Distribution with Less than Eight Experiments Installed

The likelihood that at times in the life of the EGCR several of the eight loops for experimental use will be empty or will contain graphite plugs prompted a study of the reactor power distribution under such conditions. The case in which all eight experiment tubes are present but contain no experimental assemblies was taken to be the reference case and comparisons were made with this condition. All calculations were two-group and two-dimensional, and the results are given in Table 1.1. The important observations which may be made from the data are that, when loaded with experimental assemblies, the small, 5-in.-diam loops located near the center of the core have a peaking effect on the radial power distribution, whereas the large, 9-in.-diam loops have a flattening effect. When the loops are empty, their effects are the opposite of the above because of the poisoning of the reactor by the permanent steel through-tubes.

Effect of Control Rods on EGCR Power Distribution

Previously reported studies of the EGCR power distribution¹ have been extended to include the effects of inserting various control rods into the reactor core. The principal motivation for this study was the need to determine acceptable control rod configurations for reactor operation at full power with experimental assemblies installed in the loop facilities. For this determination the four large experiment tubes were assumed to be filled with graphite, since this represents the most probable condition at the startup of the reactor and is also the condition which presents the greatest problems in control of the reactor

¹"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, pp. 4-8.

Table 1.1. Effects of Loading Experimental Assemblies into Fewer Than Eight Loops in the EGCR Core

Loop Contents ^a		Relative Reactivity, Δk	Radial Peak-to-Average Power Ratio	Power Per Loop (Mw)
Small Loops	Large Loops			
Void	Void	0	1.254	0
Void	Graphite	0.0026	1.274	0
Void	Loop tube absent	0.01532	1.1809	0
Graphite	Experimental assembly	0.0176	1.170	1.314
Experimental assembly	Graphite	0.0418	1.758	1.338
Experimental assembly ^b	Graphite ^b	0.0172 ^b	1.352 ^b	1.073 ^b

^aThe experimental assemblies considered were homogeneous graphite-U²³⁵ mixtures with a carbon-to-uranium ratio of 200 (0.33 kg of U²³⁵ per foot in small loops; 0.923 kg of U²³⁵ per foot in large loops).

^bCentral control rod fully inserted.

power distribution. The four small experiment tubes were assumed to contain a homogeneous graphite-U²³⁵ assembly with a carbon-to-uranium ratio of 200 (0.33 kg of U²³⁵ per foot).

Two-dimensional, two-group reactor calculations were performed which permitted the explicit representation of the eight experimental facilities and selected groups of fully inserted control rods. As described previously,² the cylindrical control rods were represented by equivalent rods of rectangular cross section, and an extrapolation length into the rod was specified for each group. Identification letters were assigned to the control rods as shown in Fig. 1.1. A summary of the results for the eight cases studied is presented in Table 1.2.

²Ibid., pp. 9-11.

Fig. 1.1. Identification Letters Assigned to EGCR Control Rods.

Table 1.2. Effect of the Full Insertion of Selected Control Rods on the Radial Power Distribution in the Initial 2.2%-Enriched EGCR Core

Control Rods Inserted	Radial Peak-to-Average Power Ratio	Reactivity Decrease, Δk	Average Power Per Loop (Mw)
None	1.758	0	1.338
N	1.352	0.0246	1.073
N, G, O, T, M	1.201	0.0870	0.682
G, O, T, M	1.188	0.0791	0.866
M, O	0.637	0.0400	1.144
C, P, X, L	2.149	0.0260	1.480
P	1.852	0.0068	1.370
N, O	1.622	0.0380	1.007

Because of the heat transfer limitation imposed by the available pumping power, the radial peak-to-average ratio in the reactor must be less than 1.35 when the core is operating at full power. It is clear, then, that only the second, third, and fourth cases shown in Table 1.2

represent acceptable configurations of fully inserted control rods for a uniform radial core loading.

Since only the radial power distribution was covered by the above analysis, it became important to examine the general case in which one or more rods are partially inserted into the core. The results for the cases of full insertion of selected control rods indicated that an acceptable, and perhaps required, programed motion of the rods would involve coordination of the central rod with four of the inner ring of eight control rods. Since it would be highly undesirable for the criticality of the reactor at any time to depend upon the continued operation of the loop experiments, the normal operating condition for the reactor will involve the insertion of sufficient poison by the control rods to compensate for the reactivity effect of four experiments, the operating margin, and the allowance for override of peak xenon poisoning. Using an average allowance for four experiments derived from a previous study,¹ the total allowance for the above effects can be set at a Δk of approximately 0.05. Examination of Table 1.2 shows that this amount of control could be obtained with the central rod (rod N) fully inserted and with rods G, O, T, and M approximately one-half inserted. With the central control rod fully inserted the radial peak-to-average ratio is sufficiently low at both limits for the positions of rods G, O, T, and M, and similarly, if these four rods are fully inserted, the peak-to-average ratio is sufficiently low at both limits for the position of the central rod. Since, with the central rod fully inserted, rods G, O, T, and M provide approximately as much control as the anticipated maximum reactivity effect of eight loop experiments ($\Delta k \sim 0.06$), these control rods appear to represent an attractive choice for controlling the reactor. The central control rod would be fully inserted at all times and could be considered to be holding down the operating margin and the reactivity allowance for overriding peak xenon poison.

An exact analysis of the reactor power distribution for the configuration described above would require a three-dimensional analysis of the core, but the geometry is sufficiently complex to place this calculation outside the capability of the existing computing facilities.

An analysis based on two-dimensional calculations has been developed, however, which should provide a sufficiently accurate description of the three-dimensional system. This analysis is based on the fluxes in a long, reflected reactor being separable into individual radial and axial variations over a large portion of the reactor volume. If this condition is met in each half of the EGCR core, the radial variations in these regions may be obtained by two-dimensional calculations. The three-dimensional flux distributions in the reactor may then be obtained by normalizing the appropriate radial variation with the function $P(z)$ defined by

$$P(z) = \iint P(x,y,z) \, dx dy , \quad (1)$$

where $P(x,y,z)$ is the three-dimensional power distribution normalized so that

$$\frac{\iiint P(x,y,z) \, dx dy dz}{\iiint dx dy dz} = 1. \quad (2)$$

The determination of $P(z)$ from Eq. (1) requires, of course, a knowledge of the three-dimensional power distribution, which is approximated by writing

$$P(z) \sim \int P(r,z) \, 2\pi r \, dr , \quad (3)$$

where $P(r,z)$ results from a two-dimensional calculation of the three-dimensional reactor in cylindrical form. At any point in the core,

$$P(x,y,z) = P_{T,B}(x,y) P(z) , \quad (4)$$

where $P_{T,B}(x,y)$ is the radial power distribution in either the top or bottom half of the reactor, depending upon whether the point z is above or below the mid-plane of the core.

If the condition of separability of the fluxes is sufficiently well satisfied, the principal difficulty encountered with this procedure arises in the treatment of the transition region between the top and bottom portions of the reactor, since a discontinuity in the fluxes will result at the boundary between the two regions. In the present study, with the central rod fully inserted and rods G, O, T, and M inserted to the mid-plane, the calculation that produces the function $P(r,z)$ appearing in Eq. (3) was compared with one-dimensional calculations of the top and bottom portions of the core in order to determine the width of the transition region. The fluxes were found to obey the separability condition quite well throughout the core, except in a region extending 10 in. on either side of the core mid-plane. With this determination of the width of the transition region, it is possible to perform a satisfactory smoothing of the discontinuity which occurs at the core mid-plane.

With the partial insertion of control rods, there is no unique axial power distribution in the core, the axial distribution being different in each channel. The four graphs of Fig. 1.2 show typical axial power distributions for the case described above and illustrate the smoothing operation necessary to give a continuous power variation at the core mid-plane. The axial peak-to-average power ratio for each channel is given in Table 1.3. If the axial power distributions are integrated along the length of the channels, the resulting integrals will represent an over-all radial power distribution. Tables 1.4, 1.5, and 1.6 give this radial distribution, together with the distributions in the top and bottom portions of the core, separately. Since the over-all radial peak-to-average power ratio is considerably less than the maximum allowable value of 1.35, this manner of operating the control rods is attractive from this point of view. Although the values calculated for the axial peak-to-average ratio are high, these values must be compared with those resulting from other methods of programming the control rods to determine the relative merits of each method. The variation of the axial peak-to-average power ratio with the reactivity effect, Δk , of bank insertion of the rods is given in Fig. 1.3. It may be seen from this figure that the

Fig. 1.2. Axial Power Distribution in Typical Fuel Channels with the Central Control Rod Fully Inserted and Rods G, O, T, and M Inserted to the Core Mid-plane. The four central loops contain homogeneous graphite- U^{235} experimental assemblies with a carbon-to-uranium ratio of 200. The normalization is to unity for the entire core.

Table 1.3. Axial Peak-to-Average Power Ratio in Each Channel of the Core when the Central Rod is Fully Inserted and Rods G, O, T, and M are Inserted to the Mid-Plane

y	Axial Peak-to-Average Power Ratio in Indicated Channel (x,y) ^a								
	x=4	x=12	x=20	x=28	x=36	x=44	x=52	x=60	x=68
4	1.904	1.909	1.974	1.914	1.764	1.661	1.607	1.554	1.536
12	1.909		1.857	1.799	1.713	1.649	1.601	1.559	1.553
20	1.974	1.857	1.790	1.729	1.679	1.623	1.578	1.522	
28	1.914	1.799	1.729	1.692	1.642	1.599	1.571	1.547	
36	1.764	1.713	1.679	1.642	1.618	1.588	1.580	1.548	
44	1.661	1.649	1.623	1.599	1.588				
52	1.607	1.601	1.578	1.571	1.580				
60	1.554	1.559	1.522	1.547	1.548				
68	1.536	1.553							

^ax and y are coordinates of channel in inches from center of reactor.

Table 1.4. Over-all Radial Power Distribution (Normalized to Unity) with the Central Control Rod Fully Inserted and Rods G, O, T, and M Inserted to the Mid-Plane

y	Over-all Radial Power Distribution in Indicated Channel ^a (x,y) ^b								
	x=4	x=12	x=20	x=28	x=36	x=44	x=52	x=60	x=68
4	0.788	1.037	1.150	1.194	1.224	1.171	1.051	0.908	0.809
12	1.037		1.201	1.247	1.226	1.140	1.018	0.885	0.818
20	1.150	1.201	1.262	1.249	1.184	1.075	0.944	0.828	
28	1.194	1.247	1.249	1.182	1.088	0.957	0.821	0.745	
36	1.224	1.226	1.184	1.088	0.952	0.803	0.660	0.604	
44	1.171	1.140	1.075	0.957	0.803				
52	1.051	1.018	0.944	0.821	0.660				
60	0.909	0.885	0.828	0.745	0.604				
68	0.809	0.818							

^aFour small experiment loops assumed to contain homogeneous graphite-U²³⁵ assemblies with a carbon-to-uranium ratio of 200.

^bx and y are coordinates of channel in inches from center of reactor.

Table 1.5. Radial Power Distribution (Normalized to Unity) for
Top Half of Core when Central Control Rod is Fully Inserted
and Rods G, O, T, and M are Inserted to the Mid-Plane

y	Radial Power Distribution in Indicated Channel (x,y) ^a								
	x=4	x=12	x=20	x=28	x=36	x=44	x=52	x=60	x=68
4	0.518	0.666	0.617	0.748	1.074	1.198	1.185	1.081	0.994
12	0.666		0.885	1.020	1.157	1.202	1.159	1.058	1.004
20	0.617	0.885	1.064	1.156	1.195	1.170	1.093	1.028	
28	0.748	1.020	1.156	1.194	1.162	1.077	0.967	0.905	
36	1.074	1.157	1.195	1.162	1.061	0.924	0.794	0.738	
44	1.198	1.202	1.170	0.077	0.924				
52	1.185	1.159	1.093	0.967	0.794				
60	1.081	1.058	1.028	0.905	0.738				
68	0.994	1.004							

^ax and y are coordinates of channel in inches from center of reactor.

Table 1.6. Radial Power Distribution (Normalized to Unity) for
Bottom Half of Core when Central Control Rod is Fully Inserted
and Rods G, O, T, and M are Inserted to the Mid-Plane

y	Radial Power Distribution in Indicated Channel (x,y) ^a								
	x=4	x=12	x=20	x=28	x=36	x=44	x=52	x=60	x=68
4	0.885	1.170	1.342	1.349	1.274	1.150	0.999	0.837	0.735
12	1.170		1.320	1.327	1.240	1.111	0.961	0.816	0.745
20	1.342	1.320	1.335	1.277	1.168	1.031	0.884	0.786	
28	1.349	1.327	1.277	1.185	1.056	0.907	0.763	0.681	
36	1.274	1.240	1.168	1.056	0.909	0.753	0.616	0.553	
44	1.150	1.111	1.031	0.907	0.753				
52	0.999	0.961	0.884	0.763	0.616				
60	0.837	0.816	0.786	0.681	0.553				
68	0.735	0.745							

^ax and y are coordinates of channel in inches from center of reactor.

Fig. 1.3. Variation of Axial Peak-to-Average Power Ratio with Δk for Bank Insertion of the EGCR Control Rods.

axial peak-to-average power ratio rises to about 2.44 if the bank of control rods is inserted to the depth required to give the same reactivity effect as that of the case considered ($\Delta k \sim 0.05$). At the same time, the radial peak-to-average power ratio will be the same as that for the first case shown in Table 1.2, that is, 1.758, which is considerably in excess of the maximum allowable value.

Nonuniform Neutron Absorption on the Surface of EGCR Control Rods

Peaking factors have been obtained which describe the variation of the neutron absorption rate at the tip of a partially inserted EGCR control rod and azimuthally around fully inserted rods at several positions in the core. The peaking factor for the tip of a partially

inserted control rod was determined from a two-dimensional cylindrical reactor calculation with a control rod inserted to the mid-plane at the center of the core. The axial variation of the ratio of the absorption rate in the control rod to the reactor power density at a radius some distance from the rod is nearly constant for a fully inserted rod. For a partially inserted rod the ratio is constant except within a distance of approximately 10 in. from the tip. This ratio, normalized to unity in the asymptotic region, is plotted in Fig. 1.4 and is interpreted to be the factor by which the reactor axial power density distribution should be multiplied to get a relative neutron absorption rate in a partially inserted control rod.

The azimuthal variation around several control rods was determined from the calculations summarized in Table 1.2, although the representation of the control rods by equivalent rectangular regions prevented

Fig. 1.4. Peaking Factor for Neutron Absorption Near the Tip of a Partially Inserted Control Rod. A/A_0 is ratio of perturbed to unperturbed absorption rate.

a direct determination from the results of the calculations. If the absorption on each side of the rectangle is assumed to correspond to the absorption in the corresponding quadrant of the actual cylindrical rod, the azimuthal variation may be represented by a polar histogram. These integral absorption data can then be used to determine a continuous variation by fitting each with a function of the form

$$A(\theta) = 1 + B \cos\theta ,$$

where θ is the angle with respect to the x axis of the core. The constant B is overdetermined if all the available data are used, but, if it is fixed by fitting the ratio of the absorptions in the quadrants at 0 and 180 deg, the absorptions by quadrant are given to a 1% accuracy by the analytical form for all cases. The constant B for each case examined is listed in Table 1.7. It should be noted that the magnitude of B for a specific control rod depends greatly on the positions of the other control rods.

Table 1.7. Amplitude of the Azimuthal Variation
of the Neutron Absorption Rate,
 $A(\theta) = 1 + B \cos\theta$

Rods Inserted	Rod Described	B
P, X, L, C	P	-0.1672
O, T, M, G	O	+0.0244
N, O, T, M, G	O	+0.0789
N, O	N	-0.0913
N, O	O	-0.0389

Neutron Spectra Calculations

For the purpose of calculating average cross sections, it is convenient to have the neutron spectrum represented parametrically by a

simple function. The spectrum is usually approximated as the sum of a Maxwellian distribution and a $1/E$ distribution terminated at the low energy end by a suitable joining function. In order to investigate the adequacy of this approximation and to determine the nature of the joining function, a series of spectrum calculations for different moderator temperatures and absorption cross sections has been carried out on the IBM-704. The assumption made in these calculations is that the moderator is an infinite carbon-gas medium. Spectra have been calculated for the following conditions:

Moderator Temperature (°K)	Absorption Cross Section, Σ_{2200}^a (barns/carbon atom)
300	0.05, 0.15
600	0.05, 0.15, 0.40, 0.50
1200	0.05, 0.10, 0.15

A preliminary analysis of the joining functions of the above spectra has indicated that a single joining function can be found which will adequately represent the calculated spectra. The joining function appears to be qualitatively similar to that of Campbell and Freemantle,³ but it has considerably different parameters.

Campbell and Freemantle chose for a joining function a linear rise from energy αkT to βkT and a broad maximum from βkT to μkT joining onto a $1/E$ spectrum above μkT , with $\alpha = 1$, $\beta = 3.6$, and $\mu = 5$. The spectra calculated here fit much better, however, with $\alpha = 2$, $\beta = 5$, and $\mu = 7$. These are tentative parameters and may be revised as additional spectra are calculated. A minor revision, which simplifies calculations and appears inconsequential in terms of computed reaction rates, is to make the joining function constant between βkT and μkT .

³G. C. Campbell and R. G. Freemantle, "Effective Cross Section Data for Thermal Reactor Calculations," AERE-RP/R 2031 and Supplement 1 (1957), also paper P/10, 1958 Geneva Conference.

BeO-Moderated Reactors

A series of calculations has been started to explore the nuclear characteristics of two-region, semihomogeneous, gas-cooled, BeO-moderated reactors in which the fissionable material ($U^{233}O_2$) is in a central cylindrical core and the fertile material (ThO_2) is entirely in a surrounding blanket. With 4000 beryllium atoms per uranium atom in the core, a core diameter of 70 cm was obtained. This diameter limits the thermal power output to about 100 Mw per core if reasonable limits are put on the heat transfer properties of the system. A net conversion ratio of 1.10 was obtained after making allowances for parasitic capture, leakage, and processing losses. The net conversion ratio was virtually constant as the burnup of the fuel on a continuous fueling cycle was varied from 20 to 60%. In this range, the increased parasitic loss to fission products as the burnup increased was offset by the decreased processing losses (assumed to be 0.5% of the material processed).

The beryllium-to-uranium atomic ratio of 4000, together with the assumption that 14% of the moderator is contained in the fuel elements, resulted in a high cost for replacement of BeO during fuel element refabrication. A calculation was made in which the beryllium-to-uranium ratio was reduced to 2000 and only 8.7% of the beryllium was in the fuel. In this case, the BeO replacement cost was considerably reduced, whereas the conversion ratio stayed about the same. The total thermal power per core was reduced, however, to about 40 Mw.

Further calculations will be made of multiple-region reactors in an effort to retain the high conversion ratios resulting from complete segregation of fissionable and fertile materials and, at the same time, increase the total power output to an economically attractive value.

2. REACTOR DESIGN STUDIES

EGCR Fuel Assemblies

Mechanical Design

The drawings for the Title II design of the EGCR fuel assembly have been completed, and an assembly drawing is shown in Fig. 2.1. The top and bottom spiders serve to position the rods relative to each other and to position the cluster of rods within the graphite sleeve. The top spider, which supports the fuel rods, rests on a shoulder in the graphite sleeve so that during normal fuel handling by the charge machine from the bottom of the reactor there will be no load placed on the two shear pins attached to this spider.

If it became necessary to remove the fuel from a channel by using the service machine at the top of the reactor, the assembly would be lifted by a grapple attached to the top spider and the shear pins would carry the weight of the graphite sleeve. The six radial tabs on the bottom spider are for centering purposes only and are not required to support loads other than small side loads caused by thermal gradients and the resultant bowing of the rod cluster.

In the event an assembly became "jammed" in a channel, a special grapple would be used to shear the two pins attached to the top spider and to lift the fueled part of the assembly from the reactor. In this operation the radial tabs on the bottom spider would bend and could be pulled through the graphite sleeve. Results of tests conducted at room temperature on the shear pins and bending tabs are presented in Tables 2.1 and 2.2. Tests will also be conducted at 1100°F to check the load-carrying capacity of the pins near their maximum operating temperature in the reactor. Based on the ratio of the strength of type 304 stainless steel at room temperature to its strength at 1100°F, two 0.090-in.-diam pins should withstand a lifting load of about 500 lb. The maximum lifting load to be applied to the pins at operating conditions will be about 150 lb.

Fig. 2.1. EGCR Title-II Fuel Element Assembly.

Table 2.1. Shearing Load Per Pin as
a Function of Pin Diameter

Pin Diameter (in.)	Load (lb)	Stress (psi)
0.105	515	59 400
0.101	492	61 400
0.093	436	64 200
0.093	466	68 600
0.075	314	71 100
0.072	321	78 800

Table 2.2. Load Required to Bend
Tabs and Pull Bottom Spider
Through Sleeve

Tab Thickness (in.)	Load (lb)
0.030	39
0.050	80
0.070	400

Pressure Drop and Flow Distribution

Pressure drop tests of the spacer shown in Fig. 2.1 have been completed. It was found that the pressure drop for this design was within a few per cent of that for the Title I design with 45-deg helical teeth on the spacers.¹ For this case the loss contributed by the spacers for a channel flow rate of 2400 lb/hr of helium was a total of 1.9 psi for the six fuel element assemblies in the channel. The losses for the expansion and contraction between assemblies with the new spider design are now being determined.

The tests of the flow distribution in the cluster have been initiated. Preliminary measurements at a small length-to-diameter ratio near the

¹"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, p. 29.

entrance to a cluster were compared with mass transfer data for 0- and 30-deg orientation between assemblies, and good agreement was found. It thus appears that the results of the mass transfer tests give a good indication of the velocity distribution at the entrance to an assembly. Based on the mass-transfer data there will be no severe maldistributions of flow for 0-, 30-, 60-, and 90-deg relative orientations between assemblies.

Thermal Analysis of Fuel Assemblies

Since the EGCR is to serve as a test facility for fuel materials and fuel element designs, the number of possible modes of control and therefore the number of flux and heat-generation profiles throughout the core will be quite numerous. The nominal maximum fuel element surface temperature as a function of the channel mixed-mean gas temperature and the maximum channel exit gas temperature for one mode of operation is shown in Fig. 2.2. In this case it was assumed that the central control rod was fully inserted, the four control rods in the inner ring were one-half inserted, and the remaining control rods were in the upper reflector.

In the calculations for this case it was assumed that all 232 channels in the core were loaded and that 80 of the 85 Mw of heat generated remained in the fuel elements, with the remainder being in the graphite sleeves and the graphite moderator. The coolant flow required to remove the heat generated in the core structure and to cool the control rods was considered. In order to attain the specified mixed-mean exit gas temperature of 1045°F, the temperature of the gas leaving the fuel channels must be 1065 to 1075°F. Variations of channel orificing were assumed in the calculations to establish the desired temperature conditions. Based on a mixed-mean exit gas temperature of 1075°F from the fuel channels, various orificing arrangements gave the results presented in Fig. 2.2. For the case in which all channels have an exit temperature of 1075°F, the maximum nominal surface temperature is 1445°F. This temperature will exist in only eight channels. The lowest maximum surface temperature for this case will be about 1190°F and will exist in 16 channels. The other channels will be at temperatures between these extremes.

Fig. 2.2. Maximum Fuel Element Surface Temperature Versus Mixed-Mean Exit Gas Temperature from 232 Channels of EGCR.

If the channels are orificed so that the maximum surface temperature in all channels is the same, the surface temperature is 1280°F, a reduction of 165°F. For this case the maximum exit gas temperature is 1150°F, and the minimum exit gas temperature is 965°F.

Depending upon the results of graphite oxidation tests and whether the graphite sleeves are given a protective coating, it may be necessary to limit the sleeve temperature to prevent oxidation of the graphite in the event of the maximum credible accident in which air may be circulated through the core. Again, from Fig. 2.2, it may be seen that a reasonable compromise is available for limiting both the surface temperatures of the fuel elements and the gas temperatures. In this case an upper limit of the gas temperature can be set so that part of the channels are orificed to fix the maximum surface temperature and the remainder of the channels are orificed to fix the exit gas temperature. If about 60% of the channels were orificed for gas temperature control and 40% for surface temperature control, the maximum gas temperature would be 1100°F, the mixed-mean gas temperature would be 1075°F, and the maximum fuel element surface temperature would be 1300°F.

All the fuel element surface temperatures given here are nominal in that they do not include any hot channel factors or allowances for flux peaking within the cluster, for temperature asymmetries within a channel, or for operating procedures such as fuel charging and discharging while at power. These factors will add about 250°F to the nominal fuel element surface temperature. In considering the maximum gas temperatures, an allowance of 25°F must be made for the accuracy of the exit gas temperature measurement and another 25°F for operating margin. In addition to this 50°F variation, the effect of charging and discharging fuel must be considered. When the fuel is removed from one channel in the core the power in the adjacent channels increases by 8%. The exit gas temperatures from the channels adjacent to the empty channel will increase by 40 to 50°F unless the orifice position for these channels is adjusted prior to the fuel-handling operation.

The nominal fuel element surface and gas temperatures for the highest power channel in the core are shown in Fig. 2.3. For this case the mixed mean exit gas temperature is assumed to be 1075°F, with the nominal maximum gas temperature limited to 1100°F. The sharp breaks in the surface temperature are the result of flow disturbances at the entrance to an assembly or at the mid-cluster spacers.

Heat Transfer Correlations

A considerable amount of heat transfer data taken on rod clusters at ORNL and at Allis-Chalmers has been analyzed to determine a correlation for the average heat transfer coefficient as a function of Reynolds number and axial position. A plot of the ratio of the film Nusselt number (Nu_f) to the 0.4 power of the film Prandtl number (Pr_f) versus the film Reynolds number (Re_f) is presented in Fig. 2.4. These data were obtained at Allis-Chalmers on a seven-tube bundle with a 2:1 tube-spacing ratio at a distance of 21.7 equivalent diameters downstream of the 60-deg-pad mid-cluster

Fig. 2.3. Nominal Fuel Element Surface and Gas Temperatures Along Channel 4-20, the Highest Power Channel of the EGCR.

Fig. 2.4. Heat Transfer Correlation as a Function of Film Reynolds Number for a Seven-Tube Bundle with a 2:1 Tube-Spacing Ratio at a Distance of 21.7 Equivalent Diameters Downstream from a 60-deg-Pad Mid-Cluster Spacer. Data obtained at Allis-Chalmers.

spacer shown in Fig. 2.1. Data from a series of 16 runs fall within $\pm 10\%$ of the curve of the equation

$$Nu_f = 0.0229 Re_f^{0.8} Pr_f^{0.4} ,$$

when the gas properties are evaluated at a temperature which is the average of the wall temperature and the bulk gas temperature.

The coefficient,

$$C = \frac{Nu_f}{Re_f^{0.8} Pr_f^{0.4}} ,$$

is plotted versus the length-to-diameter ratio in Fig. 2.5 for data obtained both at Allis-Chalmers and at ORNL. The tube bundle used for the Allis-Chalmers experiments had a 2:1 tube-spacing ratio, where the tube-spacing ratio is the clearance between the center tube and outer tube divided by the clearance between the outer tube and the sleeve, flat

Fig. 2.5. Heat Transfer Correlation as a Function of Length-to-Diameter Ratio.

Micarta spacers at the inlet, and a 60-deg-pad mid-cluster spacer, as mentioned above. The bulk gas flow Reynolds number was 30 000. The tubes were 0.75 in. o.d. and the sleeve was 3 in. i.d. The ORNL data were obtained for a tube bundle with 0.8-in.-o.d. tubes and a 3.83-in.-i.d. sleeve. The tube-spacing ratio was 4:1 and the bulk gas flow Reynolds number was 76 000. The mid-cluster spacer was the one-piece set-screw type described previously.² There was no spacer at the inlet to the bundle.

The two sets of data fall within $\pm 5\%$ of an average curve both upstream and downstream of the mid-cluster spacer. The upstream and downstream data of Allis-Chalmers fall within $\pm 5\%$ of their average of the two, and the ORNL upstream and downstream data fall within $\pm 10\%$ of their average. This indicates that the variation of the coefficient C of the heat transfer correlation with the length-to-diameter ratio is not a

²"GCR Quar. Prog. Rep. June 30, 1959," ORNL-2767, p. 76.

strong function of the exact inlet spacer geometry but is different for the case where the entrance is smooth and no spacer is used.

Radiative Heat Transfer in EGCR Fuel Bundle

A study of the radiative heat transfer among the seven rods and sleeve of the EGCR fuel cluster was undertaken. Since neither the graphite sleeve nor the stainless steel cladding of the rods is black to heat radiation, there results an unending number of reflections, absorptions, and reradiations between the various surfaces. Because of the complicated geometry involved and the complex heat transfer mechanisms, the study was aimed primarily at determining what effect the radiative heat transfer had on the temperature difference across one of the outer rods of the bundle.

The results of the study yielded view factors for the various surfaces that take into account emissivities, geometrical configurations, and other factors and are independent of temperature. These view factors can be used to determine the heat interchanges between the various zones in a simple fashion. One estimate of the effect of radiation on the diametral temperature difference across an outer rod of the cluster indicates that the temperature difference based on flux peaking in the outer rods may be diminished by about 20°F by radiative heat transfer.

A simplified model for studying the over-all radiative heat transfer from the seven rods to the graphite sleeve was developed that gave excellent agreement with the results obtained by considering an accurate model. In the simplified model the effective rod area was considered to be the area based on the length of a string wrapped around the seven rods. The values obtained for the heat transferred from the seven rods to the graphite sleeve by the two methods differ by about 5%. The more exact method of analysis is necessary, however, for determining temperature distributions around the individual fuel rods.

The assembly considered consisted of a 3-in.-i.d. graphite sleeve and seven 0.750-in.-o.d. rods with the outer six rods on a centerline diameter of 2 in. The results of the heat transfer calculations are

shown in Figs. 2.6 and 2.7. In Fig. 2.7 the heat transferred from surface 2 is 5.2% greater than that from surface 1.

Analytical Studies on Fluid Shifting in EGCR Fuel Bundle

Because of the differences in flow resistance in the various parallel flow paths of the EGCR fuel cluster, the coolant shifts from one region in the cross section to another as it proceeds along the rods.³ The hangers and spacers also cause abrupt changes in flow distribution by virtue of the areas they block out.

³"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 16-21.

Fig. 2.6. Radiative Heat Transfer from Seven-Rod Cluster to Graphite Sleeve for Various Values of Rod Emissivity.

Fig. 2.7. Radiative Heat Transfer from Two Surfaces of Seven-Rod Cluster to Graphite Sleeve for Various Rod Surface and Sleeve Temperatures.

A mathematical model has been developed for studying this mass shifting and predicting the temperatures of the parallel fluid streams as they progress through the reactor. The model has been translated into a code for the IBM-704 so that temperatures can be calculated for various rod spacings, hanger and spacer effects, flux distributions, and

gas flow rates, and for variations in other physical parameters. The model is based on the static pressure being constant over the entire cross section at any axial position and on considerations of energy balances. The only major assumption is that the flow in the cluster is exclusively radial.

Two cases have been studied numerically in an attempt to bracket the possible conditions in the reactor. A rod spacing that would give an inside mass flow ratio (the ratio of the mass flow in one of the interior passages of the bundle to that of the total) of 0.055 for isothermal incompressible flow at an infinite distance from a disturbance was chosen. In case 1, the flow on the inside passage was assumed to mix completely with that on the outside at every hanger and spacer. The maximum gas temperature difference between an inner and outer passage was found to be 22°F, and it occurred at the exit end of the third bundle in the direction of coolant flow. In case 2, it was envisioned that the spiders and hangers would redistribute the flow, without turbulence, in such a way that the pressure drop through the obstruction would be equal in the inside and outside passages of the obstruction. Thus, the only mixing was due to a net transport of gas between the inner and outer passages. This limited mixing model yielded a maximum gas temperature difference across the rods of 102°F at the exit end of the fifth bundle for the same axial flux distribution as for case 1.

Fission-Gas-Release Studies

A comparison of the top-loading and the inverted-loading fuel programs⁴ for the EGCR has been made based on the fuel element life code developed by Rosenthal.⁵ For assigned heat-generation rates, surface temperatures, and fuel geometry and properties, the code computes the fuel element internal temperature structure, the amount of

⁴Ibid., pp. 22-4.

⁵M. W. Rosenthal, "PANDORA - A Computer Program for Estimating Fission Gas Release and Its Effect on the Mechanical Life of Clad Fuel Elements," ORNL CF-60-5-1.

fission gas released, the fuel element internal pressure, and the cladding strain when the internal pressure exceeds the coolant pressure. The creep damage is also computed, and the calculation is terminated if the rupture strain is reached.

Average heat generation rates and surface temperatures for each of the six axial fuel channel positions were determined from the heat generation and surface temperature distributions for each of the two fuel-loading programs. A fixed, radial, fuel-to-cladding gap of 0.003 in., the largest possible within EGCR fuel specifications, was used throughout the analysis. In order to allow for hot-channel factors not included in the surface temperature distribution analyses, the mechanical property data for the cladding strain calculations were taken at a temperature 100°F higher than the maximum surface temperature for each fuel assembly position.

For the inverted-loading program and an effective fission-product noble-gas diffusion coefficient at 1400°C of 10^{-9} sec^{-1} , all assemblies achieve the desired exposure without excessive internal pressure buildup or cladding strain. The results of these calculations are summarized in Table 2.3.

Since the heat generation rates and surface temperatures for the upper half of the core are much higher in the case of top loading, only the behavior of fuel initially charged in the position of maximum power generation (position number 2, the second assembly from the top) has been examined. An effective fission-gas diffusion coefficient at 1400°C of $10^{-11} \text{ sec}^{-1}$ is required to prevent rupture of the cladding before the desired exposure is reached. For this case the internal pressure at discharge is 80 psia and the fuel exposure is 11 000 Mwd/MT of uranium. For an effective diffusion coefficient of 10^{-9} sec^{-1} , the element fails in 273 days, and for a coefficient of $10^{-10} \text{ sec}^{-1}$ failure occurs in 429 days.

The average fuel exposure at discharge is the same for the two loading programs and the deviation of the exposures of the individual assemblies from this average is also approximately equal. On the basis of this analysis, the inverted-loading program is the more desirable of

Table 2.3. Results of Inverted-Loading Fuel Program Analysis
Based on a Coolant Pressure of 300 psia
and a Fuel Exposure Period of 1040 days

$$D'_{1400^{\circ}\text{C}} = 10^{-9} \text{ sec}^{-1}$$

	Fuel Assembly					
	A	B	C	D	E	F
Initial assembly position within fuel channel	1 (top)	2	3	4	5	6 (bottom)
Heat generation rate (Btu/hr·ft)	7 000	15 100	22 500	24 700	20 900	15 400
Average surface temperature, °F	1 125	1 185	1 200	1 110	960	800
Peak surface temperature, ^a °F	1 250	1 310	1 350	1 325	1 170	980
Internal pressure at exposure period midpoint, psia	50	70	105	110	75	80
Fraction of fission-gas released at exposure period midpoint	3.0	3.0	6.3	6.9	3.1	3.0
Fuel exposure at exposure period midpoint, Mwd/MT of uranium	2 000	4 300	6 400	7 000	5 900	4 400
Channel position of fuel assembly during second half of exposure period	4	5	6	1	2	3
Internal pressure at discharge, psia	190	90	85	85	90	350
Cladding strain at discharge	0	0	0	0	0	0.00004
Fraction of fission-gas released at discharge	12.9	3.3	4.9	6.0	3.4	24.6
Fuel exposure at discharge, Mwd/MT of uranium	9 000	10 200	10 700	9 000	10 200	10 700

^aValue given is 100°F above calculated maximum surface temperature to allow for hot spots.

the two, since it results in lower fuel element surface temperatures and permits the use of UO_2 with an effective fission-gas diffusion coefficient a hundredfold larger than that required for the top loading program. Although most postirradiation annealing experiments performed thus far have yielded $D'_{1400^\circ\text{C}}$ values much lower than those assumed in this analysis, it should be noted that the samples used for this type of measurement were irradiated at low temperatures to low burnups (see sec. 4). Values of $D'_{1400^\circ\text{C}}$ which yield fractional gas-release values similar to those obtained in prototype fuel irradiations provide a better indication of the fuel behavior to be expected during EGCR operation.

The fission-gas-release code also provides a convenient method of studying the effects of fuel element design parameters on expected fuel behavior. When the $D'_{1400^\circ\text{C}}$ value which characterizes the UO_2 fuel is large enough to indicate release of more than a few per cent of the fission gases formed, the combination of a large fuel-to-cladding radial gap and a low helium fill pressure result in a large temperature drop and accelerated fission-gas release and pressure buildup. The internal pressure and cladding strain behavior of a fuel element initially charged in the bottom fuel channel position with the inverted fuel-loading program has been calculated for a series of helium fill pressures and two $D'_{1400^\circ\text{C}}$ values, assuming a fixed 0.003-in. radial gap. In the results, presented in Table 2.4, the exposure time indicated is that at the end of the last time interval before the failure strain was exceeded for those cases in which the full 1040-day exposure could not be achieved. For the largest fuel-to-cladding gap which could exist with the present EGCR fuel element tolerances (0.003 in.), Table 2.4 indicates that the helium fill pressure should be limited to a minimum of 10 psia if experimental results show that $D'_{1400^\circ\text{C}}$ values as large as 10^{-9} sec^{-1} are to be expected. Also, Table 2.4 indicates that the helium fill pressure will not be very important in determining fuel element behavior if $D'_{1400^\circ\text{C}}$ values as low as $10^{-11} \text{ sec}^{-1}$ are characteristic of the EGCR fuel pellets.

Table 2.4. Internal Pressure of Fuel Element Calculated for a Series of Helium Fill Pressures and Two Effective Diffusion Coefficient Values

Helium Fill Pressure (psia)	$D'_{1400^{\circ}\text{C}} = 10^{-9} \text{ sec}^{-1}$		$D'_{1400^{\circ}\text{C}} = 10^{-11} \text{ sec}^{-1}$	
	Final Pressure (psia)	Exposure Time (days)	Final Pressure (psia)	Exposure Time (days)
1	727	832	206	1040
3	720	858	78	1040
5	701	884	63	1040
10	763	1040	79	1040

Statistical Analysis of Hot-Spot Temperature in EGCR Fuel Assembly

A statistical method has been developed for estimating the probability that the hot spot on the EGCR fuel element will exceed a given temperature. This method has a decided advantage over simple multiplication of individual hot-spot factors, since it yields a result that does not assume that all the factors involved attain their worst values at the same spot and time.

For the analysis a realistic picture of the temperature of the fuel rod surface was drawn. The fuel element surface temperature depends on ten independent variables which have the following uncertainties:

Gas temperatures at inlet to core	$\pm 40^{\circ}\text{F}$
Macroscopic fission cross section of fuel	$\pm 2\%$
Radial peak-to-average flux ratio	$\pm 1\%$
Axial peak-to-average flux ratio	$\pm 2\%$
Cross sectional area of fuel pellets	$\pm 1.5\%$
Graphite sleeve inside diameter	$\pm 0.2\%$
Stainless steel tubing outside diameter	$\pm 0.8\%$
Nominal heat transfer coefficient	$\pm 10\%$
Observed gas outlet temperature	$\pm 25^{\circ}\text{F}$
Thermocouple error	$\pm 25^{\circ}\text{F}$

The expression for fuel rod surface temperature was expanded into a Taylor series about the mean value of each of the variables. Since the linear terms suffice to represent the temperature change that an incremental change in the independent variables will induce, the problem of finding the frequency function for the surface temperature resolves to the problem of adding ten frequency functions. Each of the ten frequency functions represents one of the linear terms in the Taylor expansion for the incremental temperature change and, for simplicity and conservatism, was considered rectangular with extremes as given above.

The addition of ten square frequency functions is an arduous task, but fortunately it was found that only five rectangular distributions, each having extremes differing by not more than a factor of 2 or 3, sufficed to yield a frequency function that closely approached Gaussian form. The other five factors could only enhance this congruity, and so it was decided to consider that the result of adding all ten distributions would yield a normal distribution with a variance (square of the standard deviation) equal to the sum of the individual variances. (The variance of a rectangular frequency function is one third the square of the difference between its extreme and mean.)

Thus, for the values quoted above, the standard deviation of the surface temperature was found to be 35.6°F. Therefore the probability that the hottest spot on the fuel element surface will be at a temperature above the indicated value is the following:

Temperature (°F)	Probability of Being Above this Temperature
Nominal	0.50
Nominal + 20	0.29
Nominal + 40	0.13
Nominal + 60	0.046
Nominal + 80	0.012
Nominal + 100	0.0025
Nominal + 120	0.00038

These probabilities were based on early flux profile determinations and preliminary estimates of tolerances, but no large changes are expected, although the values are sensitive to the methods of flux flattening and control of the reactor.

EGCR Control Rods

Cooling of the EGCR control rods is currently to be accomplished by directing helium coolant from the bottom plenum through orifices in the bottom plate located at control rod hole positions. Since the control rod holes are situated at block intersections (only four of the 25 rod holes are in the center of blocks), the coolant flow is partially diverted through leakage paths between the blocks. It has therefore been necessary to size each orifice for 450 lb/hr flow in order to insure at least a flow rate of 100 lb/hr up through the hollow control rod. With this design, a flow restriction is also required at the top of each hole to limit annular flow around the rods.

In order to minimize this core leakage, an alternate method for cooling the control rods has been investigated. This method consists of utilizing the coolant flow now provided for the control rod drive mechanism by directing a portion of this flow down the shroud and through the inside of the hollow control rod. This flow would leave the bottom of the rod and flow up around the outside of the rod and between the graphite blocks back to the top plenum. This cooling arrangement would require that (1) the top of the control rod be open to flow, (2) a relatively close fit (1/8-in. radial clearance) be maintained between the top outer surface of the rod and the inner surface of the shroud tube, (3) the perforations in the shroud tube be eliminated, and (4) separate bottom orifices be provided for cooling the large experimental tubes. In addition, the control rod orifices would have to be eliminated in the bottom plate as would the top flow restrictions. All these design changes appear to be of a minor nature and should be easy to accomplish.

The current design flow rate for rod drive cooling of 125 lb/hr (at 125°F) is estimated to be adequate to also cool the control rods. Should future operation of the reactor be changed so that this flow would not be sufficient, the external adjustment feature inherent in this system would become invaluable. Also, if this coolant system should become temporarily inoperable (an unlikely event, since a spare cooler is provided), no serious damage would occur to the control rods, although a peak surface temperature of approximately 1800°F might be attained.

A preliminary design of a control rod conforming to the top cooling requirements is shown in Fig. 2.8. This design differs from the previous one primarily in eliminating the gaps between segmented sections and in the addition of a spring for maintaining the segments in contact during operation while allowing for differential thermal expansion between the cable and the cans. The internal steel cable will provide considerable energy absorption during shock loading and, depending on results of tests to be made soon, may eliminate the need for a separate shock absorber.

Thermal Stresses in Top Head Nozzles of EGCR Pressure Vessel

A study of the stresses that will be created by thermal gradients in the nozzles at the top of the pressure vessel of the EGCR has indicated that, for reasonable assumptions regarding the properties of reflective insulation placed inside the thermal shield, the stresses will be large. In order to determine the stresses, it was necessary first to estimate the temperature structure in the nozzles. It was considered that heat would flow through the reflective insulation provided to protect the thermal shield from the hot gas leaving the core. Part of this heat, upon reaching the shield, would be removed by the cooling gas flowing between the shield and the pressure vessel. The remainder would radiate directly to the pressure vessel. The pressure vessel would dispose of the heat by convection to the coolant gas and by conduction through the nozzles. The conduction through the nozzles would, of course, cause thermal stresses.

Fig. 2.8. Segmented Control Rod for EGCR.

The temperature structure in the nozzles was therefore determined as a function of the physical constants of the system from heat balances for the pressure vessel and thermal shield. In order to reduce the number of independent variables to a reasonable level, the following quantities were held fixed at the values given:

Heat transfer coefficient for coolant, Btu/hr.ft ² .°F	2.75
Conductivity of nozzle material, Btu/hr.ft.°F	25
Conductivity of thermal barrier, Btu/hr.ft.°F	12
Outside diameter of nozzle, in.	
Special nozzles	9.5
Control rod nozzles	11.125
Inside diameter of nozzle, in.	
Special nozzles	7.75
Control rod nozzles	11.125
Pressure vessel area associated with nozzle, ft ²	
Special nozzles	0.9678
Control rod nozzles	1.256
Heat transfer coefficient in nozzle, Btu/hr.ft ² .°F	
Special nozzles	19.8
Control rod nozzles	25.7
Temperature of coolant into nozzles, °F	490
Heat generation rate in pressure vessel and thermal barrier, Btu/hr.ft ³	966
Thickness of pressure vessel, in.	4
Thickness of thermal barrier, in.	1
Emissivity of pressure vessel	0.85
Emissivity of thermal shield	0.6
Temperature of core exit gas, °F	1200

If the nozzles are treated as fixed-end cylinders, the maximum thermal stress is found to be dependent on the temperature existing where the nozzle joins the pressure vessel, which is the point where the maximum stress occurs. The pressure vessel temperatures and maximum thermal stresses in the nozzles that result from variations in the conductance of the reflective insulation for three different coolant gas temperatures are shown in Figs. 2.9 and 2.10. The negative conductances shown express the fact that, for very low pressure vessel temperatures and nozzle stresses, heat would have to go from the annulus between the shield and vessel to the upper plenum chamber while maintaining a shield temperature of below 1200°F, which is an impossible situation. It may be seen that for reasonable values of conductance of the reflective insulation the stresses are high, whereas the choice of a reasonable stress results in unrealistic insulation properties.

In Figs. 2.9 and 2.10, the coolant gas temperature is the temperature of the gas in the annulus between the thermal barrier and the pressure

Fig. 2.9. Effect of Variations in Conductance of Reflective Insulation on Thermal Stresses in Special Top Head Nozzles.

Fig. 2.10. Effect of Variations in Conductance of Reflective Insulation on Thermal Stresses in Control Rod Nozzles.

vessel. The pressure vessel temperature is that existing at the junctions of the nozzles and the vessel. In the calculations the heat transfer coefficient for the annulus was taken to be that which would be found near the top of the hemispherical head where the annulus flow would be quite small. At distances further from the center of the vessel head the annulus flow will increase because of gas entering from other nozzles, and the heat transfer coefficient will be considerably above the 2.75 assumed here. At the same time, the temperature of the gas will be greater. Therefore, although the results presented in Figs. 2.9 and 2.10 are good representations of the temperatures and stresses at the top of the vessel, there may be a considerable variation from these values at large radii. Also further analysis is required to check the

validity of using the fixed-end cylinder model to represent the reactor configuration.

If 2 in. of insulation is used, as presently specified, it appears that the thermal stress in the nozzles will be about 5000 psi. Based on the analysis presented here, two fairly simple methods for reducing the thermal gradients and therefore the stresses are evident. The insulation in the nozzles could be relocated to insulate them from the incoming coolant flow, and the flow to the nozzles near the center of the vessel could be increased and the flow to those at larger radii reduced.

Structural Investigations

Stress Analysis of Graphite Sleeves for EGCR Fuel Elements

As shown in Fig. 2.1, the EGCR fuel assemblies will consist of fuel rods mounted inside a 3-in.-i.d., 5-in.-o.d. graphite sleeve. Because of proximity to the fuel rods, large fast-neutron gradients will exist in the sleeves and will cause differential shrinkage that will induce stresses that will be important to the structural behavior of the assemblies. The exposure distribution in the graphite was given previously as the normalized exposure for a 20-year time interval plotted as a function of radius.⁶ The exposure is expressed in megawatt days per adjacent tonne of fuel in the Hanford reactors (Mwd/AT) since the shrinkage data are given in terms of this unit.

Based on the exposure data, the stresses were calculated for a fuel assembly with a power density of 7.55 Mw/T and a total burnup of 11 000 Mwd/T. The differences in mechanical properties and shrinkage perpendicular and parallel to the extrusion axis were taken into consideration. Since the longitudinal axis coincides with the direction of extrusion, the mechanical properties are the same in the radial and tangential directions. The stresses for an isotropic case were also calculated.

For the anisotropic case the shrinkage rate and modulus of elasticity were taken as 3.4×10^{-7} (in./in.)/(Mwd/AT) and 1.53×10^6 psi, respectively,

⁶"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, p. 19, Fig. 1.9.

in the axial direction, and 1.5×10^{-6} (in./in.)/(Mwd/AT) and 1.3×10^6 psi, respectively, in the radial and tangential directions. The results of the calculations are presented in Fig. 2.11, where the axial, radial, and tangential stresses, designated as σ_z , σ_r , and σ_θ , are plotted as functions of the sleeve radius. It may be seen that the maximum axial tensile stress (~ 1200 psi) occurs at the inner surface. Since the ultimate flexural strength of the sleeve is approximately 1800 psi, the maximum stress is within allowable limits.

The stresses for the isotropic case were calculated for comparison for the same shrinkage rate and modulus of elasticity as for the axial direction in the anisotropic case [3.4×10^{-7} (in./in.)/(Mwd/AT) and 1.53×10^6 psi, respectively]. As shown in Fig. 2.12, the maximum stresses are at the inner surface of the cylinder, and the axial and tangential

Fig. 2.11. Stresses Induced in Graphite Sleeves of EGCR Fuel Assemblies by Neutron Exposure Gradient Based on Anisotropic Properties.

Fig. 2.12. Stresses Induced in Graphite Sleeves of EGCR Fuel Assemblies by Neutron Exposure Gradient Based on Isotropic Properties.

stresses are equal and in tension. A comparison of Fig. 2.11 and 2.12 shows that the anisotropy reduces both the radial and tangential stresses. The axial stress is also slightly reduced.

Structural Behavior of EGCR Graphite Columns

The graphite columns in the core of the EGCR will be 19 ft 4 in. long, and they will be held in place at the top and the bottom. Pins in the bottom support plate will engage the lower ends of the columns, and a restraining lattice will position the upper ends. Three peripheral bands near the mid-plane of the core will limit the bowing of the columns as a result of nonuniform irradiation-induced shrinkage.

The top and bottom structural members and the graphite end configurations must be designed to withstand the maximum loads imposed by the peripheral bands. Since column failure may occur during the lifetime

of the reactor because of bowing restraint, the maximum lateral force exerted on a given unit is limited only by the load required for fracture. In analogy, consider a simple beam with the ends supported in the manner to be used for the graphite columns. If the beam is loaded to failure by a concentrated load at the mid-plane, the failure must occur in the region of application of the central load, and the ends must maintain their structural integrity.

A structural test was conducted on a 1/16-scale model of a column machined from AGOT graphite. The specimen was supported in a manner identical to that to be used for the reactor columns, and a transverse load was applied at the mid-plane. The deflection was measured by a dial gage mounted 1 in. from the center of the column. The specimen, the end supports, and the plane of loading are shown in Fig. 2.13. Failure occurred in the plane of load application, and the crack may be seen in the figure. At the time of failure the load was 79.3 lb, which gave a calculated flexural stress of ~2300 psi; the indicated deflection was 0.068 in. During the initial stages of the test it was found that, for a 0.047-in. deflection, a 0.004-in. deflection remained as a permanent set.

The brittle fracture of the specimen, as shown in Fig. 2.14, is characteristic of AGOT graphite. The machined ends of the specimen, shown in Fig. 2.15, were unaffected by the test, and thus it was demonstrated that the column design will fulfill the structural requirements described above.

Thermal Stresses in EGCR Pressure Vessel Support Skirt

The EGCR pressure vessel is to be supported by a cylindrical skirt welded to the vessel at the upper end and bolted to the concrete biological shield at the base.⁷ The skirt material will be the same as that of the vessel, type A212, grade B steel. When the temperature of the vessel

⁷"Experimental Gas-Cooled Reactor Preliminary Hazards Summary Report," pp. 3.22-3.26, ORO-196 (May 1959).

Fig. 2.13. Graphite Column Test Specimen Showing End Supports and Plane of Loading.

exceeds that of the concrete, thermal stresses that depend on its dimensions and the temperature distribution will be induced in the skirt. In

Fig. 2.14. Fractured Graphite Column Test Specimen Showing Broken Ends.

Fig. 2.15. Machined Ends of Fractured Graphite Test Specimen Shown in Fig. 2.14.

this case, the temperature is described by the equation

$$t = A_1 e^{Nx} + A_2 e^{-Nx} , \quad (1)$$

where x is the distance along the shell, A_1 and A_2 are constants determined from boundary conditions, and N is a parameter directly proportional to the square root of the ratio of the radial heat loss to the axial heat flow in the cylinder. Specifically,

$$N = \left(\frac{2h}{k\delta} \right)^{1/2} ,$$

where h is the radial conductance, k is the thermal conductivity of the metal, and δ is the cylinder thickness. The value of h depends on the conductivity and thickness of the insulation and the gas film coefficient at the surface. The fundamental design problem is to determine the value or values for N that yield a maximum stress of less than 20 000 psi.

In order to fully analyze the system, the skirt was divided into three segments of arbitrary length, and the N value for each segment was treated as an independent quantity. The heat flow and stress equations for the composite structure were written for specified temperatures at the two ends of the cylinder and for the temperature distributions given by Eq. (1) along each section. The studies described here are for the case with no insulation on the lower 20 in. of the skirt (bottom segment), a middle segment with 4 in. of insulation, and variable-length upper section. The insulation was assumed to be diatomaceous earth, and the temperatures of the pressure vessel and concrete shield were taken as 500 and 100°F, respectively.

After having established N values for the two lower sections, the maximum stress as a function of N was calculated for given lengths of the upper segment. The results for a constant skirt thickness of 2 in. are shown in Fig. 2.16. The maximum stress occurs at the upper end of the cylinder, except for cases where the length exceeds 12 in. and N is less than 0.008. For lengths between 12 and 17 in., the location of

Fig. 2.16. Maximum Axial Thermal Stress on Upper Segment of EGCR Pressure Vessel Support Skirt for Various Segment Lengths and Values of the Parameter N.

the maximum stress begins to move down the shell and to increase slightly for N values less than 0.008. The stresses at the skirt-to-vessel junction (dashed lines) continue to decrease and approach zero for a length of 24 in. with $N = 0.004$.

Calculations of the effect of various thicknesses of insulation on the top segment showed that it was not practical to provide enough insulation to reduce the stresses significantly (see Fig. 2.17). It was evident, however, that elimination of the thermal gradient in the immediate vicinity of the joint between the skirt and the pressure vessel would effect a major reduction in stress. It was found that the desired effect could be produced by heating the upper portion of the cylinder or it could be approximated by increasing the conductance of this portion. Since heating is impractical, the latter is preferred. The increase in conductance may be obtained by increasing the skirt thickness locally or by cladding the skirt metal in the region of the joint with a higher conductivity material.

Fig. 2.17. Variation of N with Insulation Thicknesses for Various Thicknesses of Copper Cladding on a 2-in.-Thick Steel Cylinder.

Investigations revealed that increasing the thickness of the upper section to 3 or 4 in. would not yield major reductions in the maximum stress. Therefore the effect of cladding the cylinder on both surfaces with equal thicknesses of copper was examined. Pure copper has a thermal conductivity approximately nine times that of carbon steel. The effects of various thicknesses of copper and insulation on the value of N for a 2-in.-thick steel cylinder are shown in Fig. 2.17. From these data it may be seen that the reducing effect on N decreases with increasing cladding thickness. Hence, the gain per unit thickness is relatively small when the cladding thickness exceeds $1/2$ in.

The temperature distribution and the resulting thermal stresses as a function of distance along the support cylinder if the upper 12 in. is made from 2-in.-thick steel plate clad on both sides with $1/2$ in. of pure copper and covered with 6 in. of insulation are shown in Figs. 2.18 and 2.19. Similar curves for unclad steel are shown for comparison. Since its strength is low at 500°F , the copper cladding does not change

the structural rigidity of the skirt. (Copper-clad steel of the specified dimensions is commercially available.)

The procedures required for making the weld between the skirt and the pressure vessel preclude cladding at the junction, and copper overlay

Fig. 2.18. Temperature Distribution in Support Skirt Showing Effect of Cladding Upper 12 in. with Pure Copper.

Fig. 2.19. Maximum Axial Thermal Stress in Support Skirt Showing Effect of Cladding Upper 12 in. with Pure Copper.

must be used to provide an adequate thermal bond between the pressure vessel and the clad skirt section. A joint design with a $3/4$ -in.-thick copper weld material overlay at the structural weld is shown in Fig. 2.20. At the axial welds of the steel cylinder there will be gaps in the copper cladding that will provide for differential thermal expansion between the steel and the copper.

The over-all skirt design shown in Fig. 2.21 is recommended. Although the analysis presented here was based on an idealized model with pure copper cladding, estimates show that the stress for the recommended design should not exceed 17 000 psi. The ASME Code allowable stress is 26 200 psi; therefore, an ample margin remains for mechanical stresses due to the weight of the vessel and other loadings.

Decontamination of Charge and Service Machines and EGCR Loops

Removal of Ruthenium

A satisfactory procedure for decontaminating carbon steel from two forms of ruthenium has been found. Ruthenium deposited on steel by

Fig. 2.20. Details of Joint Between Pressure Vessel and Support Skirt.

Fig. 2.21. Sketch of Recommended Joint Between Pressure Vessel and Support Skirt.

plating out of the peroxide-carbonate reagent used to dissolve particulate UO_2 was removed in a few minutes, with a decontamination factor of 65, by treatment in 10% Turco 4518 solution at $75^\circ C$. Deposits produced on mild steel by a drying and baking technique were decontaminated by a factor of only 5 by boiling for 30 min in 10% Turco 4518, but, when the contaminated steel was treated first with boiling alkaline permanganate and then with Turco 4518, the decontamination factor was 1×10^3 . Neither Turco 4501 nor 4501-A was effective for ruthenium removal. Thus a two-stage decontamination involving boiling in alkaline permanganate

and then in Turco 4518 (for carbon steel) or oxalic acid (for stainless steel) is recommended when ruthenium is a prominent contaminant.

Removal of Cerium

After exposure of stainless steel to cerium solutions, or after cerium solutions were dried and baked onto stainless steel at about 100°C, boiling with several dilute acids removed the cerium with decontamination factors of 10^3 to 10^4 . The peroxide-carbonate reagent gave a decontamination factor of only 2. Cerium which was heated on steel to several hundred degrees Centigrade was decontaminated by factors of only 30 to 100 by oxalic acid or Turco 4518, but, when the contaminated steel was boiled first with alkaline permanganate and then with oxalic acid or Turco 4518, the total decontamination factor was about 4×10^4 .

Procedure for Decontaminating Charge and Service Machines

For decontaminating the charge and service machines, an initial spray of hot detergent solution would be used to remove water-soluble contaminants and to flush away some of the UO_2 . Any large pieces of UO_2 would be mechanically removed. The gamma spectrum of the remaining activity, as recorded by a scintillation gamma spectrometer with a remote probe, would then indicate the next treatment. For the broad spectrum of activity of mixed fission products in fine, adherent UO_2 dust, the peroxide-carbonate reagent would be used first in a steam spray. The progress of the decontamination would be followed by a gamma monitor on the waste drain line. After a rinse, another gamma spectrum measurement would be taken with the probe inside the machine. Prominent ruthenium or cerium peaks would call for a spray with alkaline permanganate followed by Turco 4518; otherwise, only Turco 4518 would be used. The decontamination by this procedure should be 10^2 to 10^4 , depending on surface contacting and draining factors.

Procedure for Decontaminating Experimental Loops

The same general methods of decontamination can be used for the experimental loops as for the charge and service machines. In addition,

the experimental loops could be completely filled with solution if necessary, and strong acids could be used. Oxalic acid preparations have been found to be the best general-purpose decontaminants for a broad range of nuclides. The corrosion rate of boiling 10% oxalic acid was lowered from 0.011 ± 0.002 to 0.000 ± 0.002 mil/hr on type 347 stainless steel, without loss of decontamination effectiveness, by the addition of 3% H_2O_2 to the solution. The steel surface was left much freer of an oxide film than when treated with plain oxalic acid. The mixture rapidly dissolves both UO_2 and mild steel. An alkaline permanganate pretreatment is required for maximum effectiveness on ruthenium and cerium, as with the charge and service machines. Preliminary results indicate that, if a thin oxide scale is present, scale removal will be accomplished best if oxalic acid without peroxide is used for the first half hour and then peroxide is added to the circulating solution. Decontamination factors of 10^4 should be reached without difficulty if all surfaces are contacted and all activity is due to fission products.

3. EXPERIMENTAL INVESTIGATIONS OF HEAT TRANSFER AND FLUID FLOW

Resistance-Heated-Tube Heat Transfer Experiment

Data have been obtained on the circumferential surface temperature distributions and mean heat-transfer coefficients for two of the tubes in a seven-rod cluster arrangement with a tube inner-to-outer ligament ratio of 2:1 ($\gamma = 2$), which is the arrangement of the EGCR fuel elements. The data were obtained in the first of a series of experiments to be performed with the use of the model-3 heat transfer assembly.¹ This new series of experiments has been designated series 4. The adjustable mid-cluster spacer¹ used in the previous heat-transfer studies was replaced with a modified 60-deg-pad spacer.² A 0.007-in.-thick quartz-fiber tape electrically insulates the spacers from the tube surface. The inlet plenum geometry is identical to that used in the series 2 experiments ($\gamma = 4$).³ Since the model-3 apparatus has 0.8-in.-o.d. tubes in a 3.83-in.-i.d. channel, in contrast to the 0.75-in.-o.d. tubes and 3-in.-i.d. channel of the EGCR, the relative inner and outer flow areas⁴ are somewhat different. Specifically, for the series 4-A experiment, the outer region occupied 66% of the total free flow area (at a cross section other than that of the central spacer); whereas in the EGCR element, the outer portion is 68% of the total. Any effect of this difference should be most noticeable downstream of the central spacers.

The axial variation in the minimum and mean inside surface temperatures for tubes 4 and 6 are given in Table 3.1. In experiment 4-A, the over-all mean heat flux was 6500 Btu/hr.ft², and the average Reynolds modulus was 51 000. These conditions correspond approximately to those

¹"GCR Semiann. Prog. Rep. June 30, 1959," ORNL-2767, pp. 75-76.

²"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, pp. 27-28.

³Ibid., p. 57.

⁴The arbitrary dividing line is chosen to be the hexagon formed by connecting the centers of the six peripheral tubes.

Table 3.1. Axial Variation in Minimum and Mean Inside Surface Temperatures in Tubes 4 and 6 of Seven-Rod Cluster Used in Experiment 4-A

L/d_e	Minimum Inside Surface Temperature (°F)		Mean Inside Surface Temperature (°F)	
	Tube No.		Tube No.	
	4	6	4	6
0	254	253	258	261
5	355	358	368	376
10	397	401	408	417
15	427	428	433	442
17.9	431	430	437	445
20	269	279		285
22.5	334	348	340	353
25	375	389	383	397
30	439	452	445	458
35	474	487	478	493
38	482	489	487	495

anticipated near the inlet of an average channel in the reactor, allowing for the use of air rather than helium as the coolant gas. The data have been corrected⁵ to a common inlet air temperature of 97°F. The tube 6 mean inside surface temperatures were found to be from 8 to 14°F higher than those for tube 4; a similar difference was observed⁶ between these temperatures for tubes 4 and 6 in the series 2 experiments. Since tube 4 is closest and tube 6 farthest from the inlet region of the plenum, this difference may reflect a residual entrance maldistribution in the flow. The higher average surface temperatures in the series 4-A experiment resulted from the decreased Reynolds modulus ($N_{Re} = 77\ 000$ in series 2).

A comparison of the circumferential variation in the temperature profiles for tubes 4 and 6 is given in Fig. 3.1 for six selected axial

⁵"GCR Quar. Prog. Rep. Sept. 30, 1959," ORNL-2835, pp. 54-59.

⁶"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 65-70.

Fig. 3.1. Comparison of Circumferential Temperature Profiles for Tubes 4 and 6 at Six Axial Positions. Mid-cluster spacer at $L/d_e = 19$; series 4-A; $\gamma = 2$.

positions ranging from the inlet to the exit. The mid-cluster spacer is at $L/d_e = 19$. While the limited, preliminary data available prevent a detailed interpretation of these profiles, a phenomenological description along with some speculation as to cause is possible. Without regard to relative magnitudes, an idealized profile for this geometry is shown in Fig. 3.2; the location of the pad and webs of the mid-cluster spacer are shown. The profiles of Fig. 3.1 in the region upstream of the spacer ($L/d_e < 19$) show the same general characteristics, albeit with some notable differences. There are indications of maxima at 60, 180, and 300 deg and minima at 30, 120, 240, and 330 deg for both

Fig. 3.2. Idealized Temperature Distribution Around the Circumference of a Peripheral Rod in the Septafoil Geometry.

tubes, although they are more pronounced for tube 4 than for tube 6. A progressive counterclockwise displacement of the primary maximum and the primary minimum is observed for tube 6, and the profiles at intervening levels are consistent with this trend. A similar clockwise rotation is seen in the temperature patterns of tube 4. This would indicate that the flow is skewed with respect to the longitudinal axis of the tubes, and it possibly shows that there is specific redistribution of the flow in the entrance plenum.

The flow obstruction created by the 60-deg pad results in a complete upset in the temperature profiles in the portion of the cluster downstream of the spacer. The predominant change is the appearance of a temperature minimum in the vicinity of 180 deg, in contrast to the maximum which occurs at this location in the upper half of the cluster. This may be related to the increased turbulence in the wake of the spacer pad. No distinguishable effect of the webs is visible. Again, the profiles for tubes 4 and 6 are displaced with respect to each other. The consequences of using the pad spacer are pointed up by the comparison in Fig. 3.3 of the temperature distributions around tube 6 near the channel exit for both series 2 and 4-A experiments. As noted previously in the series 2 experiment, there is marked flattening of the profiles of Fig. 3.1 as the flow moves downstream; for example, for tube 6, the maximum-minimum temperature difference at the exit is only one-third the difference at the entrance.

Fig. 3.3. Comparison of the Effect of the Experimental Mid-Cluster Spacer and the 60-deg-pad Spacer on Circumferential Temperature Profiles Near the Exit of Tube 6.

Mass-Transfer Measurements

The study of heat transfer in the septafoil geometry by means of mass-removal measurements on naphthalene-coated rods has been continued. Using the apparatus previously described,⁷ mass-transfer factors were determined for the inlet half of the second (downstream) of two EGCR Title II-design fuel element clusters stacked within a Plexiglas tube. The experimental cluster is shown in Fig. 3.4 before insertion in the channel. The end spiders used with both clusters are shown in Figs. 3.5 and 3.6; 60-deg-pad spacers were cemented to the tubes in the mid-cluster position.

As illustrated in Fig. 3.4, the cluster contained only a single active (naphthalene-coated) rod; the naphthalene surface extended over approximately one-half the rod. By locating the active rod successively in each of the seven cluster positions and by reversing the rod to place the naphthalene region downstream of the central spacer, it was possible to measure mass removal over essentially the entire length of the cluster

⁷Ibid., pp. 70-76.

Fig. 3.4. Experimental Cluster Before Insertion in Plexiglas Channel.

at any rod position. The initial, or dummy, cluster served to establish a flow distribution at the entrance to the second cluster similar to that which will exist in the reactor. This entrance geometry will change as the relative orientation of the main cross ribs of the contiguous upper and lower spiders is varied between 0 and 90 deg by rotating the downstream cluster around its vertical axis (the dummy element remaining fixed).

Mass-removal data were obtained at 14 axial levels for four relative displacements of the main cross ribs; the Reynolds modulus was approximately 50 000. A complete description of the positions of the profilometer traverses is given in Table 3.2. The mid-cluster spacer is centered at $L/d_e = 22.5$. The calibration shifts reported earlier⁷ were satisfactorily resolved by substituting a plastic screw for the metal one in the differential transformer and by replacing one of the Sanborn channels.

Analyses of the data have been completed for all seven tubes at the $L/d_e = 3.8$ position and 0-deg relative orientation. As shown in Fig. 3.7, the naphthalene section begins $2.7 L/d_e$ above the lower spider; the mass-transfer entrance length is thus only $1.1 L/d_e$. The hydrodynamic entrance length is somewhat uncertain, since the naphthalene

Fig. 3.5. Upper Spider of Test Cluster for Mass-Transfer Studies.

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Fig. 3.6. Lower Spider of Test Cluster for Mass-Transfer Studies.

Table 3.2. Geometrical Locations of Profilometer Traverses

Rod No. ^a	Cluster Rotation ^b (deg)	Axial Position, ^c L/d_e													
		3.8	6.1	9.8	13.6	17.4	19.6	21.9	23.2	25.7	28.1	30.8	35.0	39.3	41.2
1	0	x	x	x	x	x	x	x	x	x	x	x	x	x	x
2	0	x	x	x	x	x	x	x	x	x	x	x	x	x	x
3	0	x	x	x	x	x	x	x	x	x	x	x	x	x	x
4	0	x	x	x	x	x	x	x							
5	0	x	x	x	x	x	x	x							
6	0	x	x	x	x	x	x	x							
7	0	x	x	x	x	x	x	x	x	x	x	x	x	x	x
1	30	x	x	x	x	x	x	x	x	x	x	x	x	x	x
2	30	x	x	x	x	x	x	x	x	x	x	x	x	x	x
3	30	x	x	x	x	x	x	x							
7	30	x	x	x	x	x	x	x	x	x	x	x	x	x	x
1	60	x	x	x	x	x	x	x							
2	60	x	x	x	x	x	x	x							
3	60	x	x	x	x	x	x	x							
7	60	x	x	x	x	x	x	x							
1	90	x	x	x	x	x	x	x							
2	90	x	x	x	x	x	x	x							
3	90	x	x	x	x	x	x	x							
7	90	x	x	x	x	x	x	x							

^aRod locations are as indicated on Figs. 3.5 and 3.6.^bDownstream cluster was rotated counterclockwise with respect to fixed dummy cluster.^cMeasured from beginning of tube (see Fig. 3.7).

sublimation during the course of a run causes the progressive growth of a surface discontinuity and concurrent disturbance of the boundary flow. Mean mass-transfer factors (circumferential average) for the seven rods at $L/d_e = 3.8$ are given in Table 3.3; the results have been adjusted to $N_{Re} = 50\,000$ on the basis that $j \sim N_{Re}^{-0.2}$. If it is assumed that the transfer characteristics in the septafoil geometry are described by the same empirical relationship as has been found to hold for heat or mass transfer in a tube with fully developed turbulent flow (i.e., the constant of proportionality between j and N_{Re} is 0.023), then the equilibrium value of the mass-transfer factor, j_∞ is calculated to be 0.00264. The average value for the ratio $(j/j_\infty)_M$ listed in Table 3.3 is 1.74. Boelter, Young, and Iverson,⁸ studying entrance-region heat transfer in tubes, found

⁸L. M. K. Boelter, G. Young, and H. W. Iverson, "An Investigation of Aircraft Heaters XVII; Distribution of Heat-Transfer Rate in the Entrance Section of a Circular Tube," NACA-TN-1451 (July 1948).

Fig. 3.7. Axial Location of Profilometer Traverse for Initial Analysis of Mass-Transfer Data.

Table 3.3. Mean Mass-Transfer Factors at $L/d_e = 3.8$ for 0-deg Cluster Orientation

Rod No.	j	j/j_∞
1	0.00442	1.70
2	0.00450	1.73
3	0.00466	1.79
4	0.00452	1.74
5	0.00466	1.79
6	0.00463	1.78
7	0.00433	1.67

for a similar geometrical situation (a hydrodynamic entrance of $2.8 L/d$) that the ratio $(j/j_\infty)_H$ at a heated length of 1.1 and $N_{Re} = 50\,000$ was 1.7. The agreement with the mass-transfer result, while perhaps fortuitous in view of the dissimilar configurations, is striking. This aspect of the results will be investigated further as the remaining experimental data are analyzed.

A polar plot of $j_l/\bar{j}$, where j_l is the local mass-transfer factor and $\bar{j}$ is the cluster mean (0.00453) at the $3.8 L/d_e$ level, is given in Fig. 3.8 for each of the rods. The profiles have been superimposed on a sketch of the cluster; included is an outline of the preceding upper and lower spiders. The tube surface corresponds to $j_l/\bar{j} = 1$. In general, the profiles for the six peripheral rods display a trifoliate pattern, with the maxima in the $j_l/\bar{j}$ curve corresponding to the three regions of relatively unobstructed flow around each rod. The decreased flow caused by the circumferential webs supporting the outer bosses of both the upper and lower spiders result in minima in the region of 60 to 85 deg and 275 to 300 deg. In like fashion, the mass removal at 180 deg is diminished by the radial tabs. The additional minima occurring on rods 1 and 4 at 0 deg are due to the flow disturbance of the cross rib. Correspondingly located minima are present on rod 7. There is also an indication on the central rod of reduced mass transfer in the regions of closest approach to the peripheral rods. The maxima on the outer rods at 120 and 240 deg lie partially in the wake of the outer ribs of the lower spider and reflect, perhaps, increased turbulence in this area.

A comparison between the mass-transfer and heat-transfer experimental results is made in Fig. 3.9. The heat transfer profile was obtained as the inverse of the temperature pattern of Fig. 3.1 for tube 4 at $L/d_e = 5$; the mass-transfer curve is for tube 6 at $L/d_e = 3.8$. The ordinate in Fig. 3.9 is an arbitrary transfer parameter. The magnitudes of the two curves are not drawn to the same scale. Since the entrance to the heat transfer cluster did not include an upper and lower spider combination, a direct comparison of the results of the two experiments is not possible. However, it is interesting to note that the same

Fig. 3.8. Circumferential Variation of Mass-Transfer Factors.
 $L/d_e = 3.8$; $N_{Re} = 50\ 000$; $\gamma = 2$.

Fig. 3.9. Comparison of Heat-Transfer and Mass-Transfer Circumferential Profiles for a Peripheral Rod in a Seven-Rod Cluster at a $\gamma = 2$ Spacing.

general pattern prevails. A detailed explanation of the differences in the profiles cannot be made at this time. For example, the role of turbulence in the wake of the spider-ribs in increasing mass transfer at the 120- and 240-deg locations or of wall conduction in decreasing heat transfer in these same regions has not yet been quantitatively defined.

Velocity Distribution in Septafoil Channels

A single seven-rod cluster with an entrance (lower) spider designed to provide minimum flow obstruction was used in previous studies⁹ of the velocity distribution in a septafoil channel. This arrangement, being similar to that of the model 3 heat-transfer apparatus, allowed comparison¹⁰ of the circumferential variation of the heat-transfer coefficient as calculated directly from experimental temperature measurements or from velocity measurements through a momentum-heat transfer analogy. For the EGCR fuel element, however, the massiveness of the upper and lower spiders and the existence of arbitrary relative rotational orientations of the adjacent spiders of successive clusters will create

⁹"GCR Quar. Prog. Rep. Sept. 30, 1959," ORNL-2835, pp. 63-69.

¹⁰"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 77-82.

flow patterns which differ substantially from those studied with the "idealized" channel.

In order to satisfy the need for information on flow in the EGCR channel, an apparatus was constructed which duplicated the geometry of adjacent clusters. This is shown in Fig. 3.10. The upper (downstream) cluster was instrumented with pitot sections as in the earlier investigation.⁹ The lower cluster was arranged so that it could be rotated through a complete 360-deg arc while the upper cluster remained fixed. The apparatus included means for studying the effects of spiders and mid-cluster spacers of various designs.

Spiders of three types have been considered thus far: (1) an EGCR Title I H-bar design (illustrated in Fig. 3.11) with cylindrical rather than conical bosses; (2) an EGCR Title II design with cylindrical bosses (Fig. 3.12); and (3) an EGCR Title II design with conical (chamfered) bosses (shown in Figs. 3.5 and 3.6). Alcorn et al.¹¹ studied the flow distribution with the H-bar spider at five relative orientations of the spiders (Fig. 3.13) between 0 deg (in line) and 90 deg without a mid-cluster spacer. The instrumented tube was used in the positions A, B, and O shown in Fig. 3.13, and data were obtained at three axial levels, $L/d_e = 3.9, 13.7, \text{ and } 22.2$, for $N_{Re} = 50\,000$.

Not unexpectedly, the flow field was found to be much more involved than that observed in the earlier investigations. In describing the flow, three separate features of specific importance may be extracted from the total complex: (1) the location of velocity maxima and minima, (2) the amplitude of these maxima and minima, and (3) the distribution of the bulk flow between the inner and outer¹² flow regions of the channel. The effect of spider orientation on the relative flows in the

¹¹W. R. Alcorn, J. A. Lauber, J. D. Mottley, and A. J. Robell, "Velocity Profiles in a Gas-Cooled Reactor," MIT Eng. Practice School Report KT-523 (Feb. 29, 1960).

¹²The inner and outer flow regions are defined as in the preceding section on resistance-heated-tube heat transfer experiments.

Fig. 3.10. Apparatus for Studying the Effect of Spider Orientation on the Velocity Distribution in EGCR Clusters.

(a) UPPER SPIDER

(b) LOWER SPIDER

Fig. 3.11. EGCR Title I Spiders.

(a) BOTTOM SPIDER
LOWER CLUSTER

(b) TOP SPIDER
LOWER CLUSTER

(c) BOTTOM SPIDER
UPPER CLUSTER

(d) TOP SPIDER
UPPER CLUSTER

Fig. 3.12. EGCR Title II Spiders with Cylindrical Bosses.

Fig. 3.13. Relative Orientations of Adjacent Title I Spiders as Examined in Velocity-Distribution Studies.

sector AOB is shown in Table 3.4 as the mean value of the ratio of the local velocity to the bulk average velocity for the entire channel, $(u/\bar{u})_m$, at the $3.9 L/d_e$ level. It may be observed that the minimum flow in the interior region occurs for this sector at the 60-deg orientation. In this configuration the tubes of the two clusters are in line, and the flow resistance of the outer region is at a minimum. Since the geometry of sector AOB is not typical of the other sectors for the 60-deg displacement (sector BOC and its mirror image, COD, in Fig. 3.13 differ from AOB in the extent and pattern of the flow restrictions caused by the spiders), it is not possible to derive general conclusions from the results of Table 3.4. Additional data will be obtained at a future time.

Velocity profiles at a distance of 0.15 in. from the central tube surface are given in Fig. 3.14 for the three axial levels. At the lowest level, the effect of the inlet spider is most pronounced. Minima occur near the 0- and 180-deg positions (although somewhat displaced), reflecting the presence of the main cross bar; the maxima at 60 and 240 deg derive from the relatively unobstructed flow in these areas (see Fig. 3.13). As the flow proceeds downstream, a continuous redistribution takes place with the mean flow increasing in the vicinity of the central tube. Thus, for the curves of Fig. 3.14, the circumferential mean

Table 3.4. Comparison of Mean Flow in Inner and Outer Zones of Sector AOB^a

Spider Orientation (deg)	$(u/\bar{u})_m$	
	Inner Zone	Outer Zone
0	0.90	1.11
15	0.92	1.17
30	0.95	1.12
60	0.82	1.12
90	0.95	1.11

^aSee Fig. 3.13 for sector delineation.

Fig. 3.14. Experimental Velocity Profiles Around Central Tube with Title I Spiders. Pitot-probe 0.15 in. from tube surface; 60-deg spider orientation; $N_{Re} = 50\,000$; $\gamma = 2$.

velocity ratio is 0.97 at $L/d_e = 3.9$, 1.02 at $L/d_e = 13.7$, and 1.08 at $L/d_e = 22.7$. This same pattern was observed in the profiles obtained at other radial distances from the tube surface.

It has been observed throughout the experimental investigation of the heat transfer and flow characteristics of septafoil channels that there are radial and rotational aspects to the flow. The single-cluster studies left doubt as to whether the source of these cross-flow components is in the channel entrance or is inherent in the cluster geometry. This feature has been investigated qualitatively with the Title I spiders using cotton fibers¹³ attached to the bosses of the entrance spider of the upper cluster. Seven such tufts, each a bundle of threads 12 in. long that were silicone-oil-coated to reduce static effects, were used. The results of the visual observations of thread deflections are sketched in Fig. 3.15. It may be seen that the flow is quite complex

¹³"GCR Semiann. Prog. Rep. June 30, 1959," ORNL-2769, pp. 63-71.

Fig. 3.15. Filament Visualization of Flow in Downstream Cluster with Title I Spiders.

and exhibits considerable nonparallel characteristics. For the 0-deg relative spider displacement, double reflection of the system around planes of symmetry is possible, and, with proper orientation, tubes B, C, E, and F are equivalent. A typical quadrant is shown in Fig. 3.16 with the appropriate thread patterns superimposed on tube B. The presence of a radial inflow to compensate for the fluid deficiency created in the central-tube environs by the spiders is clearly demonstrated. Again, at position 7, the flow is into the wake region behind the tabs.

In interpreting the filament convolutions, it must be kept in mind that the fibers extend far downstream ($L/d_e \sim 15$). Thus, the extent of the two-dimensional representations of Fig. 3.15 is a measure of the

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Fig. 3.16. Superposition of Filament-Visualization Patterns with Title I Spiders at 0-deg Orientation.

strength of the transverse flow; this is a maximum for the 15-deg displacement. At all orientations, rotating flow is evident.

The data for the Title II spider with both cylindrical (unchamfered) and conical (chamfered) bosses are incomplete. Circumferential traverses have been obtained for tube A at the $3.9 L/d_e$ level; spider orientation was varied from 0 deg (cross ribs in line) to 90 deg. A 60-deg-pad spacer was included in the upper cluster (see Fig. 3.10). Velocity profiles at a distance of 0.15 in. from the tube surface are given in Fig. 3.17 for the 0-deg spider displacement. The Reynolds modulus was 50 000.

The velocity profiles for both Title II spiders show similar features that can be qualitatively described in terms of the flow interferences

Fig. 3.17. A Comparison of Velocity Profiles for Peripheral Tube A with Title I and Title II Spiders. Pitot-probe 0.15 in. from tube surface; 0-deg spider orientation; $N_{Re} = 50\ 000$; $\gamma = 2$.

caused by specific portions of the spiders. The differences between the two curves are of more interest; since data are limited, these variances will be noted without an attempt at complete explanation. A peak in the velocity profile could be expected at 105 and 255 deg, corresponding to the unrestricted region between the webs of the adjacent spiders (see sketch of Fig. 3.8). Such maxima exist with the cylindrical boss spider at the anticipated positions. For the conical boss spider, however, these peaks are displaced circumferentially (50 deg) toward the outside portion of the tube. In contrast, minima appear with the chamfered spider at 120 and 240 deg (resulting from the webs of the upper spider of the inlet cluster), but they are shifted inward with the unchamfered unit. These effects may be related to the partial streamlining of the entrance spider of the test cluster and to the conical nature of the bosses (see Fig. 3.6) of the chamfered system. The lack of definable minima associated with the webs of the inlet spider at about 85 and 275 deg probably derives from the general displacement of the flow into the outer portion (near the channel wall) of the exterior flow zone. High velocities should occur over most of the inner region (~150 to 210 deg) with a slight flow suppression at 180 deg because of the cross rib supporting the central boss. Only the conical spider displays this pattern.

The results with the H-bar spider (Title I) showed a serious flow deficiency in the interior flow zone (Table 3.4) which could result in excessive surface temperatures on the inner side of the peripheral tubes and on the central tube. The effectiveness of the Title II spider in improving this situation is evident from Fig. 3.17. The mean velocity, $u/\bar{u}$, over the region from 120 to 240 deg at a distance of 0.15 in. from the tube surface and $L/d_e = 3.9$ is 0.83 for the Title I spider, 1.03 for the Title II cylindrical spider, and 1.09 for the Title II chamfered spider. This same feature is shown in Table 3.5 at five relative orientations of the spiders for flow regions defined in accord with the convention of Fig. 3.18. The results differ somewhat from those in Table 3.4 for the Title I spider (attributable to an alternative definition

Fig. 3.18. Delineation of Flow Channels in the Vicinity of a Peripheral Tube.

Table 3.5. Mean Velocities in the Vicinity of a Peripheral Tube as a Function of Spider Design and Orientation for $L/d_e = 3.9$

Flow Channel ^a	Spider Design ^b	Spider Orientation (deg)				
		0	15	30	60	90
		$(u/\bar{u})_m$				
1	I	1.06	0.99	1.07	1.08	1.11
	IIA	0.92	1.07	1.05	1.01	1.10
	IIB	1.05	1.07	1.00	1.06	1.08
2	I	0.74	0.82	0.87	0.93	0.91
	IIA	0.98	1.11	1.15	1.12	1.13
	IIB	1.07	1.09	1.13	1.13	1.16
3	I	0.84	0.79	0.89	0.79	0.91
	IIA	0.99	1.10	1.00	0.98	1.18
	IIB	1.07	1.08	1.07	1.09	1.16
4	I	1.09	1.14	1.11	1.07	1.16
	IIA	0.90	1.03	0.94	1.02	0.96
	IIB	1.11	0.95	1.13	0.97	1.00

^aFlow channel numbering convention as given in Fig. 3.18.

^bI = Title I; IIA = Title II with cylindrical boss; IIB = Title II with conical boss.

of the flow channel). However, the general conclusions remain valid. For the chamfered Title II spider, the maximum velocity occurs in the inner flow region at the 90-deg orientation.

Part 2. Materials Research and Testing

4. METALLURGICAL INVESTIGATIONS

Fuel and Moderator Materials Development

Fabrication of UO_2

Developmental work has been initiated on a nitrate recycle process for producing UO_2 powder from hard and soft UO_2 scrap from the fuel pellet fabrication process. The objective of this work is to produce UO_2 powder that is comparable to powder made from a fluoride solution. Thus far, nine batches of powder have been prepared by an experimental nitrate recycle process. A range of processing conditions was used in order to obtain information on the filterability and the physical characteristics of both the precipitate and the reduced powder. Two of the batches of powder had poor sintering characteristics, whereas the other batches had forming and sintering properties similar to those of powder derived directly from a fluoride solution.

Thermal Conductivity of UO_2

Specimens are being prepared for measurements of the thermal conductivity of UO_2 . Modifications and improvements have been made in the electrical components of the apparatus based on performance during early calibration runs. A determinate error analysis indicated that the total determinate error is $\pm 14.8\%$ at 100°C , $\pm 9.3\%$ at 300°C , and $\pm 8.9\%$ at 1000°C . Means for further reducing the possible errors are being studied.

The thermal conductivity of Armco iron was determined during calibration of the apparatus, and the data obtained are compared with the data of other investigators¹⁻⁴ in Fig. 4.1. The data are quite consistent and agree well with previously reported data up to 750°C . Few studies

¹A. C. Burr, Canadian J. Technol. 29(11), 451 (Nov. 1951).

²R. W. Powell, Proc. Phys. Soc. 46, 659 (1934).

³L. D. Armstrong and T. M. Dauphinee, Canadian J. Res. 25, Sec A, 357 (Nov. 1947).

⁴L. Silverman, J. Metals 197, 631 (May 1956).

Fig. 4.1. Thermal Conductivity of Armco Iron as a Function of Temperature.

have been made at temperatures above 750°C. The ORNL data from measurements up to 835°C, although not complete, reveal an interesting change in the thermal conductivity of iron near its Curie temperature, 768°C. A similar change has been reported in the thermal conductivity of nickel at its Curie temperature, 354°C.⁵

Mechanical Behavior of Encapsulated UO₂

The dimensional behavior of simulated EGCR fuel capsules is being investigated. The thermal conditions of the experiments were described previously,⁶ and axial expansion data for UO₂ pellets were presented.

⁵M. S. Van Dusen and S. M. Shelton, Bureau of Standards Journal of Research 12, 441 (1934).

⁶"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 87-91.

In the experiments, simulated fuel capsules are exposed to high, radial thermal gradients at inner surface temperatures up to approximately 3000°F. The capsule is surrounded by helium gas, and the pressure of the helium can be varied from slightly below atmospheric up to a maximum of 320 psi.

The data reported previously⁶ indicated that axial expansion of the UO₂ pellets is a function of the inner surface temperature. Therefore pellets with concave or "dished" ends were tested to determine whether they would expand less than pellets with flat ends. The design dimensions of the flat-end and dished-end pellets are shown in Fig. 4.2. The axial expansion data obtained for both types of pellets are compared in Table 4.1. Based on these results, pellets with one or both ends dished are being considered for use in the EGCR fuel elements in order to minimize the problem of differential axial expansion of the stainless steel capsule and the fuel column.

The fracture characteristics of UO₂ pellets during service are also of interest, since axial shifting in the capsule cannot be accommodated.

Fig. 4.2. Designs of Flat-End and Dished-End UO₂ Pellets.

Table 4.1. Comparison of Axial Expansion
of Flat-End and Dished-End UO₂ Pellets
at Various Temperatures

Maximum Inner Surface Temperature (°F)	Gross Axial Expansion (in./in. of stack height)	
	Flat-End Pellets	Dished-End Pellets
1000	0.005	0.0053
1500	0.0083	0.0081
2000	0.0122	0.0112
2500	0.0162	0.0142
2750	0.0183	0.0159

In the tests performed thus far, pellet breakup has not been severe. Typical pellets that were subjected to predicted EGCR conditions are shown in Fig. 4.3. The powder fines that result from cracks of the type shown are negligible. A longitudinal section of a typical as-tested capsule is shown in Fig. 4.4.

Total surface area measurements were made on the pellets shown in Fig. 4.3, and the results are presented in Table 4.2. It is interesting to note that the total surface area appears to be strongly dependent on the type and number of macrocracks and on the nature of the oxide surface.

Fig. 4.3. EGCR-Type Pellets After Thermal Cycling in a Simulated Fuel Capsule.

Fig. 4.4. Simulated EGCR Fuel Capsule Sectioned After Injection with Plastic. Note cracks in pellets.

Table 4.2. Results of Total Surface Area Measurements of 2.6%-Enriched, 95%-Dense, UO_2 Pellets Shown in Fig. 4.3

Time of Surface Area Measurement	Pellet No. ^a	Condition of Pellet	Total Surface Area (m^2)
Before test	7	No macrocracks	0.079
After test	6	No macrocracks	0.055
	4	One radial crack	0.099
	3	One radial crack and one crack three-fourths through wall	0.141
	5	Two radial cracks	0.154
	2	One radial crack and one circumferential crack	0.175
	1	Two radial cracks and oxidized surface	0.831

^aReading from left to right on Fig. 4.3.

It is also of particular interest to be able to predict the behavior of pellets and cladding after the cladding has collapsed around the pellets. In order to observe this interaction between the UO_2 pellets and the stainless steel cladding, two tests have been run. Prior to one test the cladding was precollapsed around the pellets by heating for

8 hr at 1900°F in argon at a pressure of 310 psi. The appearance of the precollapsed tube is indicated schematically in Fig. 4.5, based on radiographic measurements. The circumferential ridges formed in the cladding material correspond to the interfaces between adjacent pellets. The formation of the ridges is gradual, and the maximum heights are reached at the interfaces of adjacent pellets. The depressed section at the top pellet stack was formed in the open gap that resulted from the differential expansion between cladding and pellets at the isothermal collapsing temperature.

The precollapsed tube was subjected to 213 thermal cycles in which the cladding temperature ranged from 200 to 1800°F. The average heating and cooling rate was 150°F/min. Because of a malfunction in the testing device, the maximum inner surface temperature of the pellets varied from 2750 to 3200°F, and the expansion data obtained are therefore meaningless. It should be mentioned, however, that even after this high number of thermal cycles, the UO₂ powder fines collected at the bottom of the capsule weighed less than 0.1 g. There was no detectable failure of the cladding during the test.

In the second test a fuel capsule that had not been precollapsed was heated internally to elevated temperatures and then a pressure differential was applied across the tube wall. The time for uniform collapse to a point where the cladding just grasped the pellet was less than 36 hr but more than 24 hr. The average cladding temperature during the collapsing period was 1400°F, and the external pressure was 320 psi. The standard pellets used in this test were not uniform in diameter. They were larger at the ends than at the middle, and therefore the cladding first made contact at each end of the pellet during the collapsing period. The capsule was not held at 1400°F long enough for total collapse to occur.

The capsule was then thermally cycled through 106 cycles. The cladding temperatures during cycling ranged from 150 to 1430°F, and the maximum UO₂ temperature was 2200°F. The average heating rate was 120°F/min, and the average cooling rate was 140°F/min. Radial and axial

Fig. 4.5. Profile (as Shown by Radiograph) of Fuel Element Pre-collapsed by Heating for 8 hr at 1900°F in Argon at a Pressure of 310 psi.

expansion measurements were made after 1, 5, 19, 44, and 105 cycles. The expansion behavior of the collapsed capsule did not change during the test.

Examinations of the capsule after the test revealed that the cladding had not failed. The UO_2 powder fines collected at the bottom of the capsule weighed less than 0.2 g. Oblique lighting revealed what appeared to be circumferential ridges in the cladding at the pellet interfaces, but the ridges were not large enough to be located with a micrometer.

It may be concluded from the results obtained thus far in this study that (1) the expansion of a pellet within a noncollapsed tube is a function of its inner surface temperature, (2) the axial expansion of a collapsed capsule is dependent upon both the inner surface temperatures of the pellets and the cladding temperatures, (3) EGCR-prototype UO_2 pellets, although likely to crack when subjected to thermal gradients, remain in large sections and there is no shifting of material to the bottom of the capsule, and (4) the powder fines created during cracking are insignificant.

Fission-Product Release Studies

Studies of the release of Xe^{133} from samples of UO_2 irradiated for short times at low temperatures have continued with the use of the apparatus and procedures described previously.⁷ A summary of results obtained during the current period is presented in Table 4.3.

According to the diffusion model for fission-gas release, a plot of the fraction of fission gas released vs the square root of time should yield a straight line passing through the origin. Most of the samples studied to date, however, have shown a characteristic burst of fission gas followed by gas release at a rate that is linear with the square root of time. This effect is shown in Fig. 4.6 for UO_2 pellet 34-30 at 1300, 1400, and 1600°C. It was observed for pellet 34-30 that the amount of gas released in the initial burst was independent of temperature

⁷"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, p. 68.

Table 4.3. Results of Fission-Gas Release Measurements on UO₂ Pellets

Pellet Manufacturer	Sample No.	Pellet Density (% of theoretical)	BET Surface Area (cm ² /g)	Run No.	Total Test Time (hr)	Test Temperature (°C)	Total Fractional Gas Release	Release Rate Parameter, ^a D' (sec ⁻¹)
ORNL	1173-3	95.5	89.3	10	16	1400	2.43×10^{-2}	7.68×10^{-11}
Nuclear Materials and Engineering	34-30	94.4	28.6	17	6	1400	1.63×10^{-3}	2.51×10^{-13}
				18	6	1600	1.22×10^{-3}	7.75×10^{-13}
				19	6	1300	9.2×10^{-4}	6.66×10^{-14}
				20	6	1800	9.4×10^{-2}	1.10×10^{-7}
General Electric	GE-6	95.5	7.7	21	6	1400	4.6×10^{-4}	3.28×10^{-14}
Babcock & Wilcox	51-1	91.5	18.9	24	6	1800	6.5×10^{-2}	4.95×10^{-8}
Mallinckrodt	25-8	94	2.94	25	6	1400	3.1×10^{-4}	4.72×10^{-14}
				26	6	1800	6.7×10^{-3}	1.31×10^{-9}
Babcock & Wilcox	50-6	92	58.0	27	6	1400	5.6×10^{-3}	2.82×10^{-11}
				28	6	1800	1.47×10^{-1}	1.5×10^{-7}
Davison	33-5	93.5	34.2	29	9	1400	3.2×10^{-1}	6.66×10^{-12}
General Electric	GE-5	93.8	12.7	31	9	1400	1.23×10^{-2}	5.92×10^{-11}
				32	9	1600	2.92×10^{-2}	1.48×10^{-9}
				33	9	1800	9.93×10^{-2}	8.23×10^{-8}

^aCalculated from linear portion of curve of fraction released vs square root of time.

Fig. 4.6. Release of Xe^{133} from Pellet NUMEC 34-30 as a Function of Time at 1300, 1400, 1600, and 1800°C.

and that the diffusion-controlled process was predominate after 90 min. Since the release rate parameter, D' , is based on the assumption that the mechanism of release is diffusion, the D' values of Table 4.3 were computed from the linear portions of the curves.

It may be observed in Fig. 4.6 that the slopes of the curves increase with temperature over the temperature range from 1300 to 1600°C, in accordance with the diffusion model. When a sample from the same pellet was tested at 1800°C, however, a pronounced increase in the rate of release was observed. This effect is shown more clearly in Fig. 4.7. Not only is the amount released in 6 hr at 1800°C much higher than would be predicted from data obtained at lower temperatures, but also the shape of the curve is altered considerably. These observations indicate that the mechanism of fission-gas release is altered when the pellet temperature exceeds the normal sintering temperature of 1700°C.

Fig. 4.7. Release of Xe¹³³ from Pellet NUMEC 34-30 as a Function of Time at 1600 and 1800°C.

Since UO₂ is known to be volatile at temperatures above 2000°C, it was postulated that the release was occurring as a result of vaporization of UO₂ in the vacuum. As a check of this mechanism, a series of measurements was made of the weight loss of a sample of UO₂ as a function of time at 1800°C. The weight loss was found to be linear with time, and it totaled 2% in 9 hr. Since the total gas release in 6 hr was 9.4%, vaporization was ruled out as a controlling mechanism of release.

A second possible explanation for the marked increase in the rate of gas release above 1700°C is that grain boundaries may act as short-circuit diffusion paths for fission gases. As grain growth proceeds, all the gas in the volume of UO₂ through which grain boundaries have migrated could be released. In order to check this hypothesis, unirradiated samples of UO₂ from batch 34-30 were sintered at 1800°C for varying lengths of time, and the grain sizes were measured. It was found that the grain size increased from 32 to 41 μ in 6 hr at 1800°C.

The relation between the affected volume fraction, V_a , and the grain diameter, D , is given by the expression

$$V_a = 1 - \left(\frac{D_0}{D} \right)^3 ,$$

where D_0 is the initial grain diameter. Based on the measured grain sizes, the affected volume fraction was computed to be 0.52. Since this fraction is much higher than the observed gas release fraction, the rate of release of Xe^{133} at 1800°C cannot be explained on the basis of the assumption of complete release from the swept-out volume.

In order to determine whether the burst of release occurred each time the sample temperature was altered, a sample of UO_2 was tested at 1400°C and subsequently at 1600°C . The fraction of fission gas released from this sample as a function of the square root of time is shown in Fig. 4.8. It was observed that the rate of release increased with

Fig. 4.8. Release of Xe^{133} from Pellet GE-5 as a Function of Time at 1400 and 1600°C .

increasing temperature, but no burst of fission gas was observed when the temperature was raised from 1400 to 1600°C. The burst may be a result of surface oxidation of the UO_2 , high release rates from sharp corners of the pellet, or rapid release from grain boundaries.

A summary of D' values obtained to date from runs in which the gas release fraction was measured as a function of time is presented in Fig. 4.9. It may be observed that there is a greater than two orders of magnitude scatter in the D' values for the different batches of 95%-dense UO_2 . This scatter is not reduced when "true" diffusion coefficients are computed using the radius of the equivalent sphere, as calculated from BET surface area data. There is also a difference in the activation energies of samples GE-5 and 34-30, that of sample GE-5 being 100 kcal and that of 34-30, 47 kcal. Recent data obtained from Chalk River⁸ indicate that the activation energy for release of Xe^{133} from UO_2 is approximately 70 to 80 kcal. Further work will be required to clarify the mechanisms and to evaluate the activation energy for the release of Xe^{133} from UO_2 .

Graphite Fabrication

Several bodies have been formed in the continuing effort to develop high-density, low-porosity graphite. The conditions of preparation and the fired densities of these bodies are presented in Table 4.4. Apparent inconsistencies in the fired densities of bodies 9SB and 9SD and of bodies 8SE and 8SC, described previously,⁹ are due to the different methods used in mixing the binder and the graphite. The wet-mixed bodies generally developed the higher densities, but they also had a tendency to laminate. These laminations did not result from a fast firing schedule, but, rather, their presence is believed to be dependent on the forming pressure and the composition of the body. In all cases in which laminating occurred, a very slow firing schedule of 10°C/hr up to 1000°C had been used.

⁸J. A. L. Robertson, personal communication to J. L. Scott, May 25, 1960.

⁹"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, p. 97.

Fig. 4.9. Relationship Between D' and Reciprocal Absolute Temperature.

Table 4.4. Fired Densities of Various Graphite Mixtures

Binder and Graphite (both -200 mesh)	Quantity of Binder in 100-g of Graphite	Body No.	Specimen No.	Method of Mixing Binder and Graphite	Forming Pressure (tsi)	Average Fired Density (g/cm ³)
Durite SD-5143 binder ^a and Superflake 198 graphite ^b	60 g	85D	8S13	Wet	35	Laminated
			8S14	Wet	45	Laminated
			8S15	Wet	54	Laminated
	20 g	8SE	8S16	Wet	36	Laminated
			8S17	Wet	45	1.94
			8S18	Wet	54	1.94
Durite SD-5172 binder and Superflake 198 graphite	50 g	9SA	9S1	Dry	36	1.64
			9S2	Dry	54	1.65
	20 g	9SB	9S3	Dry	36	1.81
			9S4	Dry	54	1.82
	60 g	9SC	9S5	Wet	36	1.72
			9S6	Wet	45	1.73
			9S7	Wet	54	1.73
	20 g	9SD	9S8	Wet	36	Laminated
			9S9	Wet	45	Laminated
			9S10	Wet	54	1.92
Durez 16470 binder ^c and Superflake 198 graphite	60 g	10SA	10-1	Wet	36	1.69
			10-2	Wet	45	1.70
			10-3	Wet	54	1.70
	20 g	10SB	10-4	Wet	36	1.94
			10-5	Wet	45	1.94
			10-6	Wet	54	1.95
Furacarb P-3 binder ^d and graphite No. 280-H ^e	40 ml	20A	20-1	Wet	45	2.00
Furacarb P-3 binder and graphite A-625 ^e	40 ml	40A	40-1	Wet	45	1.29
Furacarb P-3 binder and graphite No. 230	40 ml	60A	60-1	Wet	45	2.02

^aBinder obtained from Borden Co.^bGraphite obtained from Superior Graphite Co.^cBinder obtained from Hooker Electrochemical Co.^dBinder obtained from Quaker Oats Co.^eGraphite obtained from Asbury Graphite Mills, Inc.

Seventy-one specimens from various bodies were tested for compressive strength, and the strengths were found to vary from 860 to 7405 psi. Bodies containing Bakelite (described previously⁹) as a binder were the weakest, whereas the series of bodies prepared with the Durite binder showed the highest compressive strength. Impregnation of specimens with Furacarb P-3 increased the compressive strength by 24 to 54%.

Reactions of UO₂ with Graphite

The rate of reaction of UO₂ with graphite that can be expected in a fuel element consisting of UO₂ dispersed in graphite is being studied as a function of temperature, UO₂ particle size, and pressure. Additional molded samples containing approximately 50 wt % -325-mesh UO₂ in a 75 wt % graphite-25 wt % carbon body have been prepared and studied by the methods described previously.¹⁰ As before, the data obtained for the reaction can be fitted by the equation

$$x = kt^{1/2} ,$$

where x is the fraction of UO₂ converted, k is the temperature-dependent rate constant, and t is the heating time in hours. The values of k as a function of reaction temperature, based on the current data, are:

Temperature (°C)	k
1200	0.110
1225	0.135
1275	0.303
1325	0.347

These values, which should be considered as being tentative, differ from those reported previously,¹⁰ because the previous temperature measurements were in error. The activation energy for the reaction appears to be about 53 kcal/mole. The major carbide phase present after the samples were quenched from the test temperatures was UC₂, but small amounts of UC were found. The composition of the uranium carbide phases present has no effect on the measurement of the reaction rate, because the rate is determined from the weight change, which is due to the release of the reaction product, CO.

Preliminary data have been obtained for the reaction between -60 +80 mesh UO₂ and graphite. The same rate equation applies, and, at 1275°C,

¹⁰Ibid., pp. 98-100.

k was found to be about 0.185. At 1325°C, k was about 0.350. One run at 1475°C showed that the reaction was essentially complete within 1 hr.

The apparatus for these determinations is being modified so that a Pt-Pt-10% Rh thermocouple can be used for the sample temperature measurements rather than an optical pyrometer.

Mechanical Properties of Graphite

Short-time tensile testing of two blocks of AGOT graphite has been completed. For the tensile tests of one block, the specimens were oriented in the direction parallel to the extrusion axis, and, for the tests on the second block, specimens oriented both parallel and normal to the extrusion axis were used. Also, three incremental-loading tests, which were essentially low-stress-rate long-time tensile tests, were performed on the first block at 78, 750, and 1100°F with a loading rate of 2 psi/hr. The results of these tests are presented in Figs. 4.10, 4.11, and 4.12.

The tensile tests of specimens from block No. 1 at room temperature and at 750°F yielded fracture stresses of 2270 to 2360 psi, which were consistent with predicted stresses. The fracture strains, however, were not so consistent; they varied from 0.165 to 0.250%. A comparison of the room-temperature data with the data obtained at 750°F shows the lack of temperature dependence of the tensile properties. The incremental-loading tests that were performed on the first block at 78, 750, and 1100°F yielded fracture stresses which varied from 1900 to 2040 psi. These values are in fair agreement with those obtained in the tensile tests, although they are slightly lower. The fracture strains again showed considerable scatter, varying from 0.165 to 0.220%, in agreement with the tensile-test data. A comparison of the results at the three temperatures shows that the fracture stresses and strains are not temperature dependent and are not time dependent, at least over the three orders of magnitude difference between the test periods of the short-time tensile tests and the long-time incremental loading tests.

The results obtained in the tests of specimens from graphite block No. 2 strongly demonstrate block-to-block variations in mechanical

Fig. 4.10. Results of Short-Time Tensile Tests of Specimens from AGOT Graphite Block No. 1. Specimens oriented parallel to extrusion axis and tested in air.

properties. Three tensile tests on specimens oriented in the parallel direction (specimens 25, 26, 27) yielded fracture stresses of only 960 to 1335 psi, with fracture strains ranging from 0.079 to 0.146%. These values exhibit a wide variation in themselves and are very low compared with the values for block No. 1. The strength of graphite in the parallel direction is usually greater than that in the normal direction; however, the tests of the specimens oriented normal to the extrusion axis (specimens 1, 8, 23, and 24) yielded fracture stresses of 1650 to 1890 psi and fracture strains of 0.299 to 0.406%. These results strongly indicated that block No. 2 contained defects which were oriented normal to the extrusion axis. Subsequent radiographs of the specimens demonstrated

Fig. 4.11. Results of Incremental-Loading Tests of Specimens from AGOT Graphite Block No. 1.

clearly the presence of low-density regions oriented normal, or nearly normal, to the axis of extrusion. Specimens 25 and 26 contained a large number of such regions; whereas, in specimen 27, the low-density regions were inclined from the normal direction. The radiographs alone are not adequate to establish whether the low-density regions are cracks, laminations, or some other type of defect.

Arc-Casting of Uranium Carbide Shapes

Uranium carbide is of interest as a fuel material for gas-cooled reactors because of its high density and high thermal conductivity. Of immediate interest is the determination of the conditions necessary for casting the UC shapes required for irradiation tests. For the required

Fig. 4.12. Results of Short-Time Tensile Tests of Specimens from AGOT Graphite Block No. 2. Specimens 25, 26, and 27 oriented parallel to extrusion axis; specimens 1, 8, 23, and 24 oriented normal to extrusion axis.

experiments, an existing arc furnace was modified to accommodate a specially designed water-cooled base plate, a copper-block mold, and a shortened, tungsten-tipped electrode. The copper-block mold was hollowed out to reduce its heat capacity, and the heat dissipation from the mold was minimized by reducing the contact area with the base plate and by flame-spraying all exterior surfaces with Al_2O_3 . These modifications were found to be necessary during preliminary runs with zirconium in place of UC. After the modifications were made, several UC cylinders, 0.400 in. in diameter and 3.25 in. long (120 to 130 g), were arc-cast. The required melting time, power density, and mechanical movements of the arc have been established.

In addition, smaller shapes, about 0.3 in. in diameter and 0.5 in. long, have been cast in a split insert-type mold. This type of mold utilizes the interface between the split mold and the copper retaining block to further reduce the rate of heat extraction from the charge. A

melting procedure for preparing these smaller shapes of UC has been established, and several acceptable cylinders have been cast.

Visual inspection of the UC shapes cast to date has shown that a slight amount of grinding will be required for smoothing the surface. Chemical analyses and metallographic studies of the shapes are in progress.

Fabrication Development of BeO and Fueled-BeO Bodies

A total of 18 pure BeO specimens ranging in density from 2.5 to 2.6 g/cm³ are being fabricated by isostatic pressing of bulk shapes and sintering to the desired density. These specimens are for use in irradiation experiments for determining the stability under long exposure to radiation of BeO suitable for use as a moderator in advanced gas-cooled reactors. They will be machined to the required dimensional specifications.

Specifications have been prepared for fueled-BeO specimens to be irradiated in the LITR. All specimens will be approximately 95% dense and will contain the fuel as a fine-grained dispersion. Fabrication studies have been conducted on two basic processes: (1) extrusion, followed by isostatic pressing, and (2) cold forming in a steel die. In these studies the fuel additions have been 30 vol % UO₂ or an equivalent amount of U₃O₈. Sintering, in all cases, has consisted of heating at 1750°C for 2 hr in hydrogen.

The BeO-U₃O₈ rods that were extruded, isostatically pressed, and sintered were comparable to the BeO-UO₂ rods described previously.¹¹ There were no apparent differences between these bodies; both fired to 95% of theoretical density and were similar in appearance. In both cases, however, warpage occurred on extruded lengths of 3 in. or more during the isostatic pressing operation. Further developmental work will be required to minimize this problem, either by improving the uniformity of the as-extruded body or by pressing much smaller lengths. The latter solution to the problem is not considered to be practical, at present, because of the additional time that would be involved in the fabrication process.

¹¹Ibid., p. 100.

Hollow, cylindrical, fueled specimens have been fabricated by cold forming in a steel die and sintering to densities of 95 to 98% of theoretical. The fuel was added as UO_2 , and the specimens were formed at green densities of 47 to 49% of theoretical. Increasing the compacting pressure to give higher green densities resulted in higher sintered densities. It was necessary to use Carbowax as a lubricant to prevent laminating of the specimens.

Similar specimens to which the fuel was added as U_3O_8 sintered to only 91% of theoretical density, and small circumferential cracks were prevalent on the outer surfaces. Such cracks are believed to be due, to a large extent, to the volume change that occurs when U_3O_8 is reduced to UO_2 ; however, cracks were not found in the extruded bodies to which the fuel was added as U_3O_8 .

Based on these experiments, it has been decided to prepare the specimens required for irradiation tests by cold pressing and sintering and to add the fuel as UO_2 .

Structural Materials Evaluation

Creep Tests of Type 304 Stainless Steel in Various Environments

Creep-rupture testing of type 304 stainless steel in argon, hydrogen, carbon dioxide, carbon monoxide, air, and nitrogen has continued, and the results obtained have further substantiated the data reported previously.¹² Further tests are planned to evaluate the creep behavior of this material in environments composed of argon and small quantities of CO and CO_2 .

Reactions of Type 304 Stainless Steel with CO- CO_2 Mixtures

It was reported previously¹³ that both CO and CO_2 oxidize type 304 stainless steel at elevated temperatures. It was determined also that CO is carburizing, whereas CO_2 is either neutral or decarburizing,

¹²Ibid., pp. 102-12.

¹³"GCR Semiann. Prog. Rep. June 30, 1959," ORNL-2767, pp. 114-5.

depending on the temperature. Further, the oxide films which formed on the metal in these atmospheres at elevated temperatures were ineffective as barriers for carbon diffusion.

If it is assumed that carburization of stainless steel by CO occurs by the formation of Fe₃C initially, the reactions necessary to account for the increase in the carbon content would be

Thus the carbon pickup by the metal would be governed by the equilibrium of CO and CO₂ with iron (reaction 1), and it should be possible to limit the carbon pickup by controlling the ratio of CO to CO₂.

In order to study these reactions experimentally, type 304 stainless steel tubing 0.002 in. thick was exposed to argon carrier gas containing 2.36 vol % CO and 0.18 vol % CO₂ (CO:CO₂ = 13.1:1) for 337 hr at 1800°F. The reaction rate under these conditions was parabolic and approximated the curve described by the equation

$$\Delta w = 0.086 t^{0.502} ,$$

where Δw is the weight gain in mg/cm² and t is the time in hours.

Becker's data¹⁴ show that the amount of carbon in equilibrium with austenite at 1800°F at a CO-to-CO₂ ratio of 13:1 is about 0.4% C. Analyses of the as-tested specimen showed a carbon content of 0.39% and, after removal of the oxide reaction products, 0.49%. These data indicate that carburization is governed by the CO-to-CO₂ ratio and is therefore controllable.

Identification of the reaction products by microscopy, electron and x-ray diffraction, and spectroscopy indicated that the bulk oxide

¹⁴M. L. Becker, "Carburizing and Graphitizing Reactions Between Iron-Carbon Alloys, Carbon Monoxide, and Carbon Dioxide," J. Iron and Steel Inst. 121, 337 (1930).

was Fe_3O_4 at the gas interface and Cr_2O_3 at the metal interface. The Cr_2O_3 appeared as green crystallites $8\ \mu$ thick that extended into the metal grain boundaries and the Fe_3O_4 . Minor quantities of other complex oxides, as well as Cr_4C , were found. It was calculated that oxidation accounted for greater than 90% of the reaction.

The formation of Fe_3O_4 as a stable reaction product was unexpected in an atmosphere that was highly reducing with respect to iron, since, according to thermodynamic considerations, iron oxides of any type would be reduced to iron under these conditions. Clearly some reaction other than the predicted reactions must account for the formation of Fe_3O_4 and Cr_2O_3 . At this time, the formation of Fe_3O_4 is thought to occur by (1) the decomposition of FeO , (2) decomposition of $\text{Fe}(\text{CO})_5$, or (3) oxidation of the spinel $\text{FeO}\cdot\text{Cr}_2\text{O}_3$ by CO_2 to form Cr_2O_3 and Fe_3O_4 .

Tube-Burst Testing of Stainless Steel

Previously reported tube-burst data¹⁵ indicated considerable differences in the times to rupture obtained for two heats of type 304 stainless steel. In order to determine whether the differences resulted from specimen geometry or variations in the material, a series of tube-burst tests was conducted on standard 6-in.-long, 0.750-in.-o.d., 0.020-in.-wall specimens fabricated from Superior Tubing Co. heat No. 23999X. Preliminary tests of 2.5-in.-long specimens had indicated times to rupture of about twice those obtained from tests on standard specimens from a different heat of material.

The results of tests at 1500°F on standard 6-in.-long specimens from two sources are presented in Table 4.5. It may be seen that the times to rupture and the strains at rupture measured for the Superior Tubing Co. material far exceed those measured in these tests for tubing produced by Ray Miller, Inc. The chemical compositions of both heats were checked, but the differences that were found were not sufficient to explain the variations in strength. Additional tube-burst tests of specimens from

¹⁵"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 115-6.

Table 4.5. Results of Tube-Burst Tests of Standard 6-in.-
Long Type 304 Stainless Steel Specimens Tested in the
As-Received Condition at 1500°F in Air

Tangential Stress (psi)	Superior Tubing Co. Material		Ray Miller, Inc., Material	
	Time to Rupture (hr)	Maximum Tangential Strain (%)	Time to Rupture (hr)	Maximum Tangential Strain (%)
6000	487	10.0	150	7.0
6000	517	15.0		
5500	690	11.7	225	
5000	1025	15.0	360	6.0
4000	>2300 ^a		1100	4.5

^aTest continuing.

the Superior tubing, both in-pile and out-of-pile, are in progress at 1300°F.

Investigations of the effect of environment on the time to rupture of seamless type 304 stainless steel capsules has continued. The results obtained thus far are presented in Table 4.6.

Tube-burst tests have also been conducted on annealed tubes previously collapsed around 0.700-in.-o.d. inserts. Two specimens, one with a mid-plane spacer, were subjected to an external pressure of 300 psi at 1800°F for 8 hr to produce uniform collapse. The burst tests were then performed in air at 1500°F with a tangential stress of 4600 psi. The specimen without a spacer ruptured in 1748 hr, whereas the time to rupture of the tube with the spacer was 1037 hr. This decrease in time to rupture did not seem to be due to the presence of the spacer, however, since the failure occurred outside the area affected by the brazing operation.

A wrinkle in the tube wall was deliberately produced during collapsing of the tubing on a third specimen. The time to rupture of this specimen, tested at the conditions given above, was 1824 hr. Since stainless steel inserts were used in this specimen, the increase in time

Table 4.6. Results of Tube-Burst Tests of Seamless,
0.020-in.-Wall, Type 304 Stainless Steel Capsules
in Various Environments

Specimen Treatment Prior to Testing	Test Environment	Test Temperature (°F)	Tangential Stress (psi)	Time to Rupture (hr)
As-received	Air	1500	1600	9146
	Argon		6500	197
	Argon		5000	795
	Argon		4300	1057
	Argon		3800	1443
Annealed at 2150°F for 2 hr	Argon	1500	3500	387
	Argon		4000	176
Annealed at 1950°F for 1/2 hr	CO		5500	615
	CO		5500	505
Annealed at 2150°F for 2 hr	CO	1600	4000	28
	Argon		3000	70

to rupture may have been due, in part, to self-welding of the tube wall to the inserts during collapsing. Additional tests are being performed with inserts coated with aluminum oxide.

Investigations of Mechanical Properties of Columbium

Tests have been initiated for evaluating the serviceability of columbium and columbium-base alloys as a fuel-element-cladding material in gas-cooled reactors using helium as a coolant. The temperatures at which such systems can operate are limited if the choice of structural materials is restricted to the currently available nickel- and iron-base alloys. Columbium possesses several properties which make it exceptionally attractive for structural applications in the temperature range of 1000 to 1400°C. In addition to its refractory nature, columbium is of interest to nuclear technology because of its low capture cross section for neutrons.

Among its other favorable qualities are its ductility and weldability and the ease with which it can be fabricated even at room temperature. Another property which increases the attractiveness of this material is the high melting point, 1499°C, of its major oxide, Cb_2O_5 . This is to be contrasted with the properties of the oxide of molybdenum, MoO_3 , which melts at 793°C and is volatile.

Columbium has the distinct disadvantage of being highly reactive with contaminants such as oxygen, nitrogen, water vapor, and hydrogen at moderate temperatures. At temperatures above 1000°C, reactions with nitrogen and carbon occur at an appreciable rate. These reactions produce compounds such as nitrides, oxides, and hydrides that may be partially soluble in the metal, depending upon the temperature. The presence of these impurities, either as a second-phase precipitate or as a soluble interstitial impurity, greatly alters the physical and mechanical properties of pure columbium. It is with the latter effect that the present studies are concerned. This work has been presented in detail elsewhere.¹⁶

Effect of Oxygen. A series of columbium bend specimens ($1.25 \times 1.00 \times 0.040$ in.) that contain various amounts of oxygen have been prepared and tested in bending at room temperature. These specimens were annealed for 2 hr in vacuum at 1300°C to recrystallize the material. The oxygen additions were made by annealing the material for various times at 400°C in oxygen at a pressure of 1 atm. Some of the samples were tested after this contamination treatment and others were given a subsequent 2-hr anneal in vacuum at 1300°C for homogenization before testing.

Representative results of these tests are given in Table 4.7. The data indicate that oxygen strengthens columbium without seriously reducing its room-temperature ductility until oxygen levels of the order of 3000 ppm are reached. The bend area of specimen No. 33, which had an oxygen content of 1500 ppm, showed evidence of severe cold working, but

¹⁶H. E. McCoy and D. A. Douglas, "Effect of Various Gaseous Contaminants on the Strength and Formability of Columbium," Presented at the Columbium Metallurgy Symposium, Sagamore Conference, June 9-10, 1960 (to be published).

Table 4.7. Effect of Oxygen on the Room-Temperature Bend Properties of Columbium from Heat 238135-B

Specimen No.	Treatment of Specimens After Annealing for 2 hr in Vacuum at 1300°C	Impurity Analysis (ppm)			Proportional Limit (psi)	Maximum Deflection (in.)	Stress at Maximum Deflection (psi)	Specimen Behavior
		O ₂	N ₂	H ₂				
22	As annealed	340	48	<1	29 600	0.25	59 500	Did not crack
11	Annealed 3 hr at 300°C in O ₂ , homogenized, cooled rapidly	410	33	<1	30 350	0.25	59 500	Did not crack
18	Annealed 4 hr at 400°C in O ₂ , homogenized, cooled rapidly	500	36	<1	33 900	0.25	64 500	Did not crack
32	Annealed 8 hr at 400°C in O ₂ , homogenized, cooled rapidly	780	39	<1	40 900	0.25	80 300	Did not crack
41	Annealed 65 hr at 400°C in O ₂ , homogenized, cooled rapidly	3000	41	9	67 500*	0.012	67 500	Cracked
20	Annealed 4 hr at 400°C in O ₂ , homogenized, cooled slowly	400	33	3	34 500	0.25	62 100	Did not crack
7	Annealed 8 hr at 400°C in O ₂ , homogenized, cooled slowly	480	36	4	36 300	0.25	60 400	Did not crack
1	Annealed 16 hr at 400°C in O ₂ , homogenized, cooled slowly	580	31	4	40 500	0.25	69 700	Did not crack
31	Annealed 24 hr at 400°C in O ₂ , homogenized, cooled slowly	900	29	6	48 500	0.25	86 000	Did not crack
33	Annealed 41.1 hr at 400°C in O ₂ , homogenized, cooled slowly	1500	36	6		0.25	132 000	Did not crack
27	Annealed 4 hr at 400°C in O ₂	470	32	4	35 200	0.25	60 000	Did not crack
3	Annealed 8 hr at 400°C in O ₂	440	33	4	38 200	0.25	59 400	Did not crack
9	Annealed 16 hr at 400°C in O ₂	440	27	4	32 550	0.25	58 700	Surface cracked
44	Annealed 24 hr at 400°C in O ₂	450	29	4	34 000	0.25	55 500	Surface cracked
43	Annealed 41.1 hr at 400°C in O ₂	1000	32	6	34 000	0.25	61 400	Surface cracked

*Stress associated with brittle fracture.

no cracks were formed, as may be seen in Fig. 4.13. The last series of five specimens listed in Table 4.7 was not homogenized after oxidizing. The analytical results for this series are probably in error, since some of the surface material was removed before the specimens were analyzed. Although the proportional limit did not appear to be raised significantly by the presence of the oxide film, surface cracks of the type shown in Fig. 4.14 formed because of the heavy concentration of oxygen near the surface.

If the results of the bend tests on the homogenized specimens are plotted as proportional limit vs oxygen concentration, two straight lines are obtained. The data for the series that was rapidly cooled fall on a straight line having a slope of 390 ppm O₂/10 000 psi, and the data for the series that was slowly cooled are best represented by a line having a slope of 360 ppm O₂/10 000 psi. The furnace-cooled specimens had slightly higher proportional limits than the specimens containing equivalent amounts of oxygen which were rapidly cooled. This result is probably due to the precipitation of columbium oxide in the slowly cooled specimens, although all specimens appeared to be single phase under the optical microscope.

Some data have been obtained that illustrate the effect of oxygen on the tensile properties of columbium at 982 and 1010°C. These data are presented in Table 4.8. The 1010°C data were obtained from 0.375-in.-diam rod specimens that were contaminated with oxygen, tested in creep

Table 4.8. Tensile Properties of 0.375-in.-diam
Columbium Specimens from Heat 599-1

Average Oxygen Concentration (ppm)	Test Temperature (°C)	Yield Strength (psi)	Ultimate Strength (psi)	Strain at Rupture (%)
300	982	3400	6500	63
580	982	7300	8800	50
670	1010	7000	a	31
1700	1010	9500	a	17

^aData not obtained.

Fig. 4.13. Columbium Annealed 41 hr in Oxygen at 400°C, Homogenized by Annealing 2 hr in Vacuum at 1300°C, and then Tested in Bending at Room Temperature. Specimen No. 33. Etchant: H₂O, HF, HNO₃, H₂SO₄. 100X.

for 400 hr at 3500 psi, and then step loaded to rupture; hence, the yield strength values are only approximate. All these specimens had oxygen concentration gradients between the outer surface and the center, and therefore the data of Table 4.8 represent trends rather than absolute values.

Effect of Nitrogen. A series of columbium bend specimens (1.25 × 1.00 × 0.040 in.) that contain various amounts of nitrogen have been prepared and tested in bending at room temperature. These specimens were annealed 2 hr in vacuum at 1300°C to recrystallize the material. Nitrogen additions were made by annealing at 1200°C for various lengths of time

Fig. 4.14. Columbium Annealed 41 hr in Oxygen at 400°C and then Tested in Bending at Room Temperature. Specimen No. 43. Etchant: H₂O, HF, HNO₃, H₂SO₄. 100X

in nitrogen at a pressure of 1 atm. Some of the specimens were given a subsequent 2-hr anneal in vacuum at 1300°C for homogenization before testing. The results of a series of tests on these bend specimens are presented in Table 4.9. The data indicate that, although nitrogen increases the strength of columbium, concentrations of the order of 1000 ppm seriously reduce the ductility.

A series of tensile specimens was also contaminated with nitrogen by the technique described above. The room-temperature tensile properties of these specimens are presented in Table 4.10. These data also show the increase in strength and decrease in ductility which results from high nitrogen concentrations.

Table 4.9. Effect of Nitrogen on the Room-Temperature Bend Properties of Columbium from Heat 238135-B

Specimen No.	Treatment of Specimens After Annealing for 2 hr in Vacuum at 1300°C	Impurity Analysis (ppm)			Proportional Limit (psi)	Maximum Deflection (in.)	Stress at Maximum Deflection (psi)	Specimen Behavior
		O ₂	N ₂	H ₂				
22	As annealed	340	48	<1	29 600	0.25	59 500	Did not crack
46	Annealed 2 hr in N ₂ at 1200°C, homogenized	410	460	4	63 300	0.25	109 200	Did not crack
10	Annealed 8 hr in N ₂ at 1200°C, homogenized	500	1200	1	70 300	0.145	144 000	Cracked
17	Annealed 24 hr in N ₂ at 1200°C, homogenized	470	2400	5	66 200*	0.135	108 500	Cracked
13	Annealed 48 hr in N ₂ at 1200°C, homogenized	320	3100	8	73 100*	0.036	114 800	Cracked
24	Annealed 65 hr in N ₂ at 1200°C, homogenized	500	5000	8	56 200*	0.009	58 200	Cracked
42	Annealed 2 hr in N ₂ at 1200°C	350	400	4	64 100	0.25	89 500	Did not crack
6	Annealed 8 hr in N ₂ at 1200°C	400	1250	2	70 300	0.029	79 600	Cracked
38	Annealed 24 hr in N ₂ at 1200°C	440	2700	13	64 000*	0.010	67 000	Cracked
8	Annealed 48 hr in N ₂ at 1200°C	340	4400	8	55 500*	0.009	59 300	Cracked
4	Annealed 65 hr in N ₂ at 1200°C	520	5400	13	35 200*	0.006	47 800	Cracked

*Stress associated with brittle fracture.

Table 4.10. Effect of Nitrogen on Room-Temperature Tensile Properties of Columbium from Heat 238135-B

Specimen No.	Treatment of Specimens After Annealing for 2 hr in Vacuum at 1300°C	Impurity Analysis (ppm)			Proportional Limit (psi)	Ultimate Strength (psi)	Rupture Strain (%)
		O ₂	N ₂	H ₂			
25	As annealed	340	48	<1	19 100	28 300	16.0
20	Annealed 4 hr in N ₂ at 1200°C, homogenized	430	2300	6	32 300	59 600	5.0
21	Annealed 7.3 hr in N ₂ at 1200°C, homogenized	650	2900	7	43 600*	43 600	0.0
17	Annealed 4 hr in N ₂ at 1200°C	400	2300	6	28 200*	28 200	0.0
24	Annealed 7.3 hr in N ₂ at 1200°C	780	2700	7	32 800*	32 800	0.0

*Stress associated with brittle fracture.

A duplicate series of tensile specimens contaminated with nitrogen was tested at 982°C. The results of these tests are given in Table 4.11. Although nitrogen additions of the order of 2000 ppm increase the proportional limit by a factor of 3 over that for essentially pure columbium, such additions do not reduce the rupture ductility at 982°C as seriously as was observed at room temperature. However, the specimens tested at 982°C had heavy surface cracks perpendicular to the tensile axis.

Effect of Hydrogen. A series of 0.040-in.-thick columbium tensile and bend specimens that contain various amounts of hydrogen have been prepared and tested at room temperature. Hydrogen additions were made by annealing the specimens in hydrogen at a pressure of 1 atm for 2 hr at various temperatures. This 2-hr annealing period did not appear to be sufficient time for equilibrium to be reached at the lower temperatures. A specimen annealed 18 hr at 500°C showed a weight gain that corresponded to a pickup of 45.8 cm³ of H₂/g of Cb. This value agrees well with the data of Sieverts and Moritz,¹⁷ who obtained a value of 47.4 manometrically.

¹⁷A. Sieverts and H. Moritz, "Solubility of Hydrogen and Deuterium in Columbium, with Review of Ta-H and V-H," Z. anorg. allgen. Chem. 247, 124-30 (1941).

Table 4.11. Effect of Nitrogen on the Tensile Properties at 982°C of Columbium from Heat 238135-B

Specimen No.	Treatment of Specimens After Annealing for 2 hr in Vacuum at 1300°C	Impurity Analysis (ppm)			Proportional Limit (psi)	Ultimate Strength (psi)	Rupture Strain (%)
		O ₂	N ₂	H ₂			
26	As annealed	340	48	<1	2 550	6 900	31.0
19	Annealed 4 hr in N ₂ at 1200°C, homogenized	430	2300	6	6 900	10 300	18.0
22	Annealed 7.3 hr in N ₂ at 1200°C, homogenized	650	2900	7	10 100	12 600	17.0
18	Annealed 4 hr in N ₂ at 1200°C	400	2300	6	7 000	9 500	9.5
23	Annealed 7.3 hr in N ₂ at 1200°C	780	2700	7	10 200	11 800	15.0

The results of room-temperature bend and tensile tests on these samples are given in Tables 4.12 and 4.13, respectively. Although several apparent anomalies exist in the data, it is felt that Figs. 4.15 and 4.16 represent the general effect which hydrogen has on the room-temperature mechanical properties of columbium. Hydrogen additions of up to approximately 200 ppm raise the proportional limit, whereas additions of greater than 500 ppm seriously embrittle the material and result in rupture at much lower stresses. The analytical data in Tables 4.12 and 4.13 point out the temperature range over which the hydride is formed by the 2-hr annealing treatment in hydrogen. Large quantities of hydrogen are picked up at 500 and 600°C, and serious embrittlement results. Above 600°C, only small amounts of hydrogen are adsorbed by the specimen, and the material is again ductile.

The data also point out that large quantities of hydrogen are absorbed even if a specimen is cooled very rapidly through the temperature range of 500 to 600°C in a hydrogen environment. The ability of columbium to pick up hydrogen from environments containing water vapor is also indicated.

Table 4.12. Effect of Hydrogen on the Room-Temperature Bend Properties of Columbium from Heat 238135-B

Specimen No.	Treatment of Specimens After Annealing for 2 hr in Vacuum at 1300°C	Impurity Analysis (ppm)			Proportional Limit (psi)	Maximum Deflection (in.)	Stress at Maximum Deflection (psi)	Specimen Behavior
		O ₂	N ₂	H ₂				
22	As annealed	340	48	<1	29 600	0.25	59 500	Did not crack
16	Annealed 2 hr in H ₂ at 100°C	360	42	5	28 150	0.25	53 500	Did not crack
25	Annealed 2 hr in H ₂ at 200°C	560	68	29	29 600	0.25	58 900	Did not crack
15	Annealed 2 hr in H ₂ at 400°C	410	28	12	38 000	0.25	57 400	Did not crack
29	Annealed 2 hr in H ₂ at 500°C	360	130	1900	26 700*	0.008	38 600	Cracked
14	Annealed 2 hr in H ₂ at 600°C	450	130	1500	28 050*	0.050	52 000	Cracked
35	Annealed 2 hr in H ₂ at 700°C	440	43	32	44 400	0.25	70 300	Edge cracked
19	Annealed 2 hr in H ₂ at 800°C	330	41	140	38 500	0.25	65 200	Edge cracked
2	Annealed 2 hr in H ₂ at 900°C	450	65	42	34 000	0.25	65 600	Did not crack
50	Annealed 2 hr in H ₂ at 1000°C, quenched in H ₂	410	79	730	33 400*	0.030	62 200	Cracked
51	Annealed 2 hr in Ar-2% H ₂ O at 500°C, quenched in this environment	700	36	13	35 200	0.25	64 800	Surface cracked

*Stress associated with brittle fracture.

Fig. 4.15. Effect of Hydrogen on Room-Temperature Bend Properties of Columbium.

Fig. 4.16. Effect of Hydrogen on Room-Temperature Tensile Properties of Columbium.

Table 4.13. Effect of Hydrogen on the Room-Temperature Tensile Properties of Columbium from Heat 238135-B

Specimen No.	Treatment of Specimens After Annealing for 2 hr in Vacuum at 1300°C	Impurity Analysis (ppm)			Proportional Limit (psi)	Ultimate Strength (psi)	Rupture Strain (%)
		O ₂	N ₂	H ₂			
25	As annealed	340	48	<1	19 100	28 300	16.0
7	Annealed 2 hr in H ₂ at 100°C	340	32	16	17 300	27 900	18.8
8	Annealed 2 hr in H ₂ at 200°C	390	36	3	21 500	32 800	17.2
9	Annealed 2 hr in H ₂ at 400°C	360	24	6	19 500	31 200	13.3
10	Annealed 2 hr in H ₂ at 500°C	440	91	740	10 900*	12 500	0.0
11	Annealed 2 hr in H ₂ at 600°C	210	64	630	10 000*	15 100	0.0
14	Annealed 2 hr in H ₂ at 700°C	600	54	56	18 100	37 800	13.3
12	Annealed 2 hr in H ₂ at 800°C	530	34	130	25 700	30 400	4.0
13	Annealed 2 hr in H ₂ at 900°C	330	32	21	18 100	35 000	14.1
15	Annealed 2 hr in H ₂ at 1000°C, quenched in H ₂				8 500*	8 500	0.0
16	Annealed 2 hr in H ₂ -2% H ₂ O at 500°C, quenched in this environment				27 000	35 800	13.5

*Stress associated with brittle fracture.

Hydrogen has also been shown to affect the high-temperature creep properties of columbium. The creep rate is greatly accelerated in an environment in which hydrogen is present, whether as pure hydrogen or as water vapor. However, hydrogen does not seem to reduce the creep ductility of columbium at elevated temperatures.

Studies of Pressure Vessel Steels

Studies are continuing in an effort to evaluate and determine the preirradiation behavior of parent plate and weld-heat-affected zones in

type SA-212, grade B, steel. The testing of drop-weight specimens prepared from 4-in.-thick plate has been completed. Information obtained from these tests indicates that the NDT (nil-ductility transition) temperature at the surface is 10°F; at the one-quarter thickness, it is 18°F; and, at the midsection, 30°F.

A total of 150 Charpy V-notch specimens to be used in radiation-damage studies of heat-affected zones have been prepared at the Rensselaer Polytechnic Institute. One-half these specimens were heated to a maximum temperature of 2400°F under conditions simulating the thermal cycle associated with a weld-energy input of 50 kj/in. The remainder were heated through the same cycle under conditions associated with an energy input of 100 kj/in. After cooling, all specimens were stress relieved at 1150°F for 4 hr.

Manufacturing and Inspection Methods Development

Fabrication of EGCR Title I Fuel Elements

Fifty prototype EGCR Title I fuel elements have been assembled in order to demonstrate fabricability of the design. One-half the elements were fabricated in the Welding and Brazing Laboratory of the ORNL Metallurgy Division, as described previously,¹⁸ and the remaining 25 were assembled by the Welding and Brazing Specialty Shop of the ORNL Engineering and Mechanical Division.

Fuel Element Inspection Methods

The development of techniques for inspecting the EGCR fuel elements is essentially complete. Much of the developmental work was accomplished during assembly tests on the 50 prototype fuel elements referred to above and was described previously.^{19,20} Only six of the 350 capsules tested were rejected, and these were rejected by the helium leak test.

¹⁸"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 123-8.

¹⁹"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, pp. 85-90.

²⁰"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 128-9.

Graphite Sleeve Inspection

Eddy-current inspections of the graphite sleeves of EGCR design that were supplied by three vendors were completed, and the test results were evaluated by metallographic examination. Forty-one of the 50 sleeves supplied by the National Carbon Company were inspected, and 36 (88%) were rejected. The only discontinuities found were crack-like and occurred on both the inner and outer surfaces of the sleeves. Many of the cracks could be attributed to laminations present in the extruded stock from which the sleeves were machined. The method of manufacture used by the National Carbon Company is illustrated in Fig. 4.17a. A large extruded block of graphite was cut into quadrants from which the sleeves were machined. The laminations, which were parallel to the surface in the extrusions, extended to the surfaces of the sleeves and appeared as cracks.

Of the 50 sleeves supplied by the Speer Carbon Company, 17 (34%) were rejected. With one exception, all discontinuities examined appeared to be inclusions of low-density graphite. It is postulated that graphite and binder accumulated on the mixing apparatus, hardened, and chipped off into the mix. Since such particles hardened before extrusion, they were of lower density than the surrounding graphite. Speer Carbon Company produced these sleeves by extruding oversized rods and machining the rods to sleeves of the required dimensions, as indicated in Fig. 4.17b. Laminations parallel to the longitudinal surface do not project to the surface and produce cracks when the extruded rod is machined. No laminations were noted in the metallographic sections examined.

Of the 50 sleeves supplied by the Great Lakes Carbon Company, 46 (92%) were rejected. During metallographic examination of areas containing discontinuities, only one crack was found. The remaining areas contained low-density inclusions of the type described above. The sleeves were manufactured as illustrated in Fig. 4.17b.

The basis upon which sleeves were rejected was the presence of an eddy-current indication as large as that produced by a 0.100-in.-deep machined notch. In general, the cracks examined were approximately twice

Fig. 4.17. Methods for Manufacturing Graphite Sleeves.

as deep as indicated by the eddy-current inspection. This can be attributed to unfavorable orientations of the cracks and the fact that electrical contact is made between the faces of a crack. The reference notch is more favorably oriented and has no electrical contact across its faces. A sleeve with a crack extending two-thirds the length is shown in Fig. 4.18. Several cross sections of the crack are shown.

Because of the random nature of the low-density inclusions, poor correlation was obtained between eddy-current signal strength and inclusion size. One inclusion, a portion of which has fallen out, is shown in Fig. 4.19.

Welding of EGCR Experimental Loop Through-Tubes

The EGCR experimental loops include tubes approximately 60 ft long that pass vertically through the core of the reactor. A design has been proposed for the installation and maintenance of these stainless steel

Fig. 4.18. Eddy-Current Inspection Trace of Graphite Sleeve with a Crack Extending Along Two-Thirds the Length.

Fig. 4.19. Low-Density Inclusion on Inner Surface of EGCR Graphite Sleeve.

through-tubes that requires welding remotely controlled from a distance of 10 to 12 ft in both the overhead and downhand positions. A schematic drawing of the joint to be welded is shown in Fig. 4.20. The developmental work under way on procedures for making the welds is based on the inert-gas-shielded consumable-electrode process. A conventional welding gun with a pull-type wire feed has been mounted on an automatic carriage so that the gun can be remotely operated in both the overhead and downhand positions in relation to a fixed linear test joint.

Both fillet and edge welds were evaluated from the joint design standpoint. The edge weld was selected on the basis of shrinkage considerations and accessibility, and test plates with both a 120-deg

Fig. 4.20. Schematic Drawing Showing EGCR Experimental Through-Tube Welds That Are to Be Made by a Remotely-Controlled Process.

included angle and a 90-deg included angle were prepared. In all tests, the 90-deg included angle minimized arc wandering and provided the more desirable cross-sectional geometry for a single-pass weld.

Since the overhead weld was considered to be the more difficult of the two welds, efforts were initially concentrated on determining the welding conditions for that type of weld. Overhead welds made with three variations of the welding conditions are shown in Fig. 4.21. As may be seen, the conditions were varied from those which promote drop transfer (the least penetration) to those which promote spray transfer (deep

(a) DROP TRANSFER.

(b) DROP PLUS SPRAY
TRANSFER.

(c) SPRAY TRANSFER.

Fig. 4.21. Weld Penetration Obtained on a Type 347 Stainless Steel Joint with Different Types of Metal Transfer Through the Welding Arc. (a) Drop transfer. (b) Drop plus spray transfer. (c) Spray transfer. Ethchant: 10% chromic-electrolytic. 3.5X.

penetration). The welds were made in the overhead position on two 1/4-in.-thick type 347 stainless steel plates that were beveled to obtain an included angle of 90 deg.

The effect on weld head geometry of aligning the torch with respect to the center of the joint is shown in Fig. 4.22. The photographs of Fig. 4.22 indicate that satisfactory welds can be made even with a misalignment of the torch with respect to the center of the joint of as much as 1/8 in.

Suitable conditions for both overhead- and downhand-position welding have been determined for linear joints, and specimens for concentric-tube welding experiments are being machined.

Reactor Vessel Penetrations

Procedures have been under study for making the joints at the various burst-slug-detection tube and thermocouple penetrations through the EGCR pressure shell. The developmental work has been essentially completed, and the progress made during this reporting period is discussed below.

Fig. 4.22. Effect of Torch Alignment with Respect to the Center of Type 347 Stainless Steel Joint. (a) Torch aligned. (b) Torch $\frac{1}{16}$ -in. off center alignment. (c) Torch $\frac{1}{8}$ in. off. Etchant: 10% chromic-electrolytic. 3.5X.

Burst-Slug-Detection Tube Penetrations. A full-size mockup of one quadrant of an EGCR burst-slug-detection nozzle assembly was fabricated in accordance with the procedures described previously.²¹ A 1-in.-thick stainless steel plate was rolled to a 2-ft-diam cylinder, 29 tube penetrations were made, the adapter nozzles were welded on, and the tubes were welded to the adapters. The completed assembly is shown in Fig. 4.23.

Every effort was made in the fabrication of the mockup to simulate conditions to be expected in the construction of the actual EGCR components. The adapter-to-plate welds were deposited in the downhand position, and the tube-to-adapter fillet welds were deposited with the tubes in the horizontal fixed position. Although welder accessibility was very limited, the welds were made with conventional welding torches.

Thermocouple Penetrations. A mockup of a thermocouple penetration assembly was also fabricated in accordance with the designs developed previously.²¹ The mockup includes 30 thermocouple penetrations of a 1-in.-thick stainless steel plate. Photographs of the partially completed and completely assembled mockup are shown in Figs. 4.24 and 4.25.

²¹"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 132-5.

Fig. 4.23. Mockup of Burst-Slug-Detection Tube Penetrations of EGCR Pressure Vessel.

As in fabrication of the burst-slug-detection tube mockup, every effort was again made to simulate conditions to be expected in the construction of the actual EGCR components. The nozzle-to-plate welds were deposited in the downhand position, and the thermocouple sleeve-to-adapter fillet welds were deposited in the horizontal fixed position. The thermocouple-to-sleeve joints were made by an induction-brazing procedure, a modification of which should be feasible for the actual construction operation. The close proximity of the thermocouples severely limited welder accessibility, but conventional, commercially available equipment was adequate for the application.

Welding of Dissimilar Metals

Welding of Ferritic to Austenitic Steels. Efforts are continuing in the development of procedures for welding ferritic steels to stainless

Fig. 4.24. Partially Completed and Assembled EGCR Thermocouple Penetration Mockup.

steels for such EGCR applications as the stainless steel-to-carbon steel joints in the primary coolant outlet piping. A procedure has been prepared for metal-arc welding of ASTM A-212 carbon steel plate to type 304 stainless steel plate in thicknesses ranging from $3/8$ in. through 2 in. in the flat (1G) position. In addition, most of the data have been obtained for the preparation of an all-position procedure for joining these materials. The International Nickel Company BP-85 electrode that was used in the development of the procedure has satisfactory deposition characteristics, freedom from porosity, and freedom from microfissuring. The general soundness of the welds is illustrated in Fig. 4.26, which shows a group of side-bend specimens.

Fig. 4.25. Completed EGCR Thermocouple Penetration Mockup.

A procedure is being developed for joining ASTM-A-398-D steel (2 1/4% Cr, 1% Mo) plate to type 304 stainless steel plate with BP-85 electrode. A small quantity of AISI type 502 steel (5% Cr, 0.5% Mo) has also been received, and welding studies on this material have been initiated.

Weld test plates of several materials (each 20 in. long and 1 in. thick) have been fabricated for room- and elevated-temperature mechanical properties studies. The fabrication of tubular dissimilar-metal weld specimens of two sizes for thermal-cycling studies is also under way.

Joining of Aluminum to Stainless Steel. Difficulty was encountered in the fabrication of a recent in-pile capsule requiring a joint between aluminum and stainless steel. The joint was designed to be fabricated

Fig. 4.26. Side-Bend Specimens of ASTM A-212 Carbon Steel Joined to Type 304 Stainless Steel with BP-85 Weld Metal Showing General Soundness of the Joints.

by flashwelding copper to the aluminum, brazing the copper to a stainless header, and welding of the aluminum to an aluminum tube. However, the brazing operation caused overheating and embrittlement of the copper-to-aluminum joint.

A test joint was prepared by plating the aluminum with nickel and then silver brazing the nickel to a stainless header. The joint was leaktight and remained so after bending. In a tensile test, the joint failed in the aluminum tube.

Advanced Fuel Element Fabrication

Interest has been expressed in EGCR-type fuel elements containing an internal pressure of approximately 0.1 atm of helium instead of helium at atmospheric pressure in order to extend the fuel capsule life substantially and yet provide enough helium to impart good heat-transfer

characteristics. Conventional tungsten-arc inert-gas-shielding welding procedures are not applicable in this reduced pressure range because of arc instability, and electron-beam welding procedures require vacuums lower than the desired 0.1 atm.

A so-called "Arvac" process for making welds in the desired pressure range is being developed, and an apparatus has been constructed for investigating the welding variables as applied to sealing a capsule. Fusion welds of apparently good quality have been made on type 304 stainless steel in helium in the pressure range from 250 to 1×10^{-3} mm Hg. Continuous fusion welds have not been attainable in this pressure range by other welding techniques.

Beryllium Investigations

Reactions of Beryllium with CO₂

Four tests have been run to investigate the reactions of beryllium with CO₂. In two of these tests, -200 mesh beryllium powder was heated at approximately 700°C in CO₂ at a pressure of 1 μ for 350 and 1000 hr, respectively. In the other two tests the temperature was 850°C, and the heating periods were 250 and 1000 hr. Tests run at the same temperature gave reproducible results. The observed oxidation rates are approximated by the following equations:

$$W_{720^{\circ}\text{C}} = 6.78 t^{0.24}$$

and

$$W_{850^{\circ}\text{C}} = 2.8 t^{0.347} ,$$

where W is the weight gain in $\mu\text{g}/\text{cm}^2$ and t is time in hours. The fact that the rate equation for 850°C predicts a lower rate of oxidation than that for 720°C is explained by the fact that sintering of the beryllium powder took place at the higher temperature and thus reduced the effective area of the specimen.

A comparison of the weight gains after 10 000 hr at 700°C is presented in Table 4.14, as calculated from the rate equations published by several investigators.²²

Table 4.14. Oxidation of Beryllium as Determined by Several Investigators^a

Investigators	Metal Source	Atmosphere	Weight Gain after 10 000 hr at 700°C (mg/cm ²)	Remarks
Gulbransen and Andrew	Brush Beryllium Co., 98.85% Be, hot pressed	O ₂ at 7.6 mm Hg	1.385	No thermal cycling
Williams and Munro	Extruded, French, flake Be	CO ₂ at 1 atm ^b	14.4	Thermal cycling
Antill and Higgins	Rolled, French, flake Be	CO ₂ at 1 atm	2.124	
ORNL	Brush Beryllium Co., QMV -200 mesh powder	CO ₂ at 1 μ Hg	0.062	No thermal cycling

^aAll but ORNL data obtained from ref. 22.

^bThe CO₂ used in this case probably contained some water vapor.

Compatibility of Beryllium with Stainless Steel in NaK Environment

The experimental assembly designed for irradiation studies of beryllium-canned UO₂ rods in the ORR pool incorporate NaK as a heat-transfer medium, and therefore it has been necessary to investigate the compatibility of beryllium with stainless steel, the outer capsule material, in a NaK environment. The results of 500-hr compatibility tests and descriptions of these test systems were presented previously.²³ A series of similar 1000-hr tests have been completed at temperatures

²²J. E. Antill, "Oxidation of Beryllium in the Absence of Irradiation," AERE-M-435 (May 1959).

²³"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 137-8.

of 600, 700, and 800°C. A gettering operation was carried out at the test temperature that reduced the oxygen content of the NaK from 70 to approximately 50 ppm.

The weight change that occurred in the 1000-hr tests paralleled those of the 500-hr tests but were of greater magnitude. The results of both series of tests are compared in Table 4.15. The weight gains of the type 304 stainless steel coupon specimens were approximately equal to the weight losses of the beryllium specimens held in contact with them.

Metallographic examinations revealed a reaction layer on the stainless steel coupons held in contact with beryllium coupons during the 1000-hr tests. The extent of the reaction was dependent on the test temperature. The layer was less than 0.0005 in. thick on specimens tested at 600°C and about 0.010 in. thick on those tested at 800°C. X-ray analyses indicated that the reaction layer was composed primarily of a NiBe compound.

Procurement of Beryllium Tubing

In order to evaluate beryllium as a fuel-cladding material for future gas-cooled reactors, beryllium tubing is being obtained from

Table 4.15. Results of Beryllium-NaK-Type 304 Stainless Steel Compatibility Tests

Test Temperature (°C)	Weight Change (mg/in. ²)					
	Beryllium Cylinder		Beryllium Coupon		Type 304 Stainless Steel Coupon	
	In 500 hr	In 1000 hr	In 500 hr	In 1000 hr	In 500 hr	In 1000 hr
600	-0.66	-0.01	Nil	Nil	Nil	Nil
700	-1.7	-1.8	-1.1	-1.4	+1.1	+1.7
800	-6.2	-10.9	-7.9	-8.4	+7.9	+7.8

foreign and domestic fabricators on purchase order and subcontract bases. The status of the purchase orders placed with the U. S. Brush Beryllium Company, the British Chesterfield Tube Company, and the French firm Pechiney for beryllium tubing representative of their best-quality commercial tubing is summarized in Table 4.16.

Table 4.16. Status of Beryllium Tubing Purchase Orders

Vendor	Fabrication Method	Size Ordered	Total Amount Ordered (ft)	Total Amount Received (ft)	Cost (\$/ft)
Brush Beryllium Company (U.S.)	Machined from hot- pressed block	0.300 in. i.d., 0.040 in. wall	125	128	36
	Machined from warm- extruded rod	0.300 in. i.d., 0.040 in. wall	125	67	36
	Machined from hot- extruded rod	0.300 in. i.d., 0.040 in. wall	125	105	36
	Warm-extruded to size	0.300 in. i.d., 0.040 in. wall	125	4	36
Chesterfield Tube Company (British)	Extruded and bore lifted	0.300 in. i.d., 0.040 in. wall	100	100	57
	Extruded and bore lifted	0.300 in. i.d., 0.040 in. wall	400		37
Pechiney (French)	Not known	0.520 in. i.d., 0.040 in. wall	137	103	26
		0.300 in. i.d., 0.040 in. wall	500	384	19

A technical report describing the methods employed by Chesterfield Tube Company to size and finish extruded beryllium tubing has been obtained from Superior Tube Company, Chesterfield's U. S. agent. Typical samples of tubing were supplied with the report.

Fabrication of Tubing

A status report has been received from Nuclear Metals, Inc., describing the work done on a subcontracted investigation of the effect of extrusion variables on the quality and properties of beryllium tubing. The work thus far has involved studies of the effects of extrusion temperature, flow shape, and reduction ratio on the production of

small-diameter tubing (0.400 in. o.d., 0.030 in. wall). Extrusions were carried out at temperatures of 1600 to 1950°F by either a filled-billet technique or by the standard technique of extruding over a mandrel; in either case, the beryllium was clad with a steel jacket.

Preferred orientation studies, burst and crush tests, and dimensional measurements were used as means of evaluating the tubing produced. In thin-walled filled-billet extrusions, the amount of preferred orientation appeared to increase with increasing extrusion temperatures and reduction ratios. The orientation in heavy-walled filled billets was unaffected by the reduction ratio. Billets extruded over a mandrel at a high reduction ratio show strong preferred orientation. In all cases except the heavy-walled filled billets, the texture consisted of basal planes normal to a tangent to the tube and parallel to the extrusion direction. The crush and burst tests gave somewhat erratic results but indicated that the highest strength tubing was obtained by extrusion of filled billets at high reduction ratios (23X) and at a temperature of approximately 1800°F.

Beryllium Tubing Evaluation

Evaluation of the beryllium tubing that has been received from American, British, and French sources is progressing. Nondestructive inspection is being accomplished by using low-voltage radiography, pulse-echo and resonance ultrasonics, penetrants, and eddy currents.

Tubing from Brush Beryllium Company. The 103 lengths of 0.300-in.-i.d., 0.040-in.-wall tubing machined from hot-pressed block and 56 pieces of similar size tubing machined from hot-extruded rod at the Brush Beryllium Company have been examined. The penetrant examination has indicated a few, shallow pinholes on all pieces, with the greater concentration being in the hot-extruded material. Radiography indicated a few, very tiny, high-density particles and localized wall thickness variations (a cyclic effect in the longitudinal direction) on much of the tubing. In addition, several small pits were found in the tubing fabricated from the hot-extruded rod. A few circumferential gouges were present along the inner surface.

The resonance ultrasonic technique which was used to measure the wall thickness rejected 53 of the 159 pieces because they did not comply with the $\pm 20\%$ tolerance. Most of the rejected tubing was both above and below the tolerance at the mid-plane of the tubing length. The ends of the tubes were much more concentric.

During eddy-current examination with a 0.006-in.-deep notch as a reference standard, all the tubing produced indications exceeding that from the reference notch. It is believed that many of the indications may have been due to the high-density particles and the localized dimensional variations.

Tubing from Pechiney. Twenty pieces of the 0.300-in.-i.d. tubing and 58 pieces of the 0.520-in.-i.d. tubing received from Pechiney have been inspected. Penetrant inspection revealed scattered pits and pinholes throughout the tubing. Three longitudinal cracks which completely penetrated the tubing wall were found. Series of short circumferential cracks of varying severity were detected in 32 of the 0.520-in.-i.d. tubes. Typical cracks are shown in Fig. 4.27. A rough measurement indicated that these cracks were 0.002 in. deep or deeper. The cracked areas will be examined further.

Radiographic examination revealed a larger amount of high-density material than was found in the Brush Beryllium Company tubing. In addition, a large number of circumferential pits were found that were estimated to be about 0.002 to 0.004 in. deep.

The pulse-echo ultrasonic examination gave six crack-like indications. Three of these had previously been revealed by penetrants. The resonance ultrasonic examination rejected two of the 0.300-in.-i.d. tubes (10%) and 13 of the 0.520-in.-i.d. tubes (22%) because of wall thicknesses below the allowable 20% tolerance.

In the eddy-current examinations, the "noise" level was much below that of the Brush Beryllium Company tubing, but many indications greater than the 0.006-in. reference notch were present. Most of the indications came from areas containing inner-surface pits.

Fig. 4.27. Typical Transverse Cracks in 0.520-in.-i.d. Beryllium Tubing from Pechiney.

Tubing from Chesterfield Tube Company. Radiographic examination of a few pieces of tubing received from the Chesterfield Tube Company revealed the presence of much high-density material, which must be removed before detailed examinations can be made. Attempts to measure the wall thickness by ultrasonic techniques have been unsuccessful, probably because of the rough, pickled outer surface. The rough surface has also presented difficulties in making sensitive penetrant inspections. Tapering of the outer surface has prevented eddy-current inspections, because of the need for a close-fitting encircling coil, and has caused problems in the use of the pulse-echo ultrasonic technique.

Beryllium Joining

The use of beryllium as a cladding material for advanced gas-cooled reactor fuel elements depends to a large extent on the development of suitable joining techniques. Development work is proceeding on various types of joining techniques, including high-temperature brazing, diffusion

bonding, fusion welding, and upset welding. The development work also includes the application of these methods to tubular fuel element fabrication and the investigations of the suitability of the various types of joints for extended elevated-temperature service.

Brazing. In an effort to develop suitable high-temperature beryllium brazing alloys for service in helium, approximate flow points have been determined for fifty experimental brazing alloys, and wettability and flowability tests have been conducted. These alloys studied were varying compositions in the following alloy systems:

1. Ag-Ti-Be	6. Pd-Al-Be	11. Cu-Ti-Zr-Be
2. Ag-Zr-Be	7. Au-Ni-Be	12. Pd-Ti-Al-Be
3. Cu-Ti-Be	8. Au-Ta-Ni	13. Au-Ti-Ni-Be
4. Cu-Zr-Be	9. Au-Ti-Ni	14. Au-Ta-Ni-Be
5. Pd-Ti-Be	10. Ag-Ti-Zr-Be	

The most promising alloys found to date are listed in Table 4.17, together with their flow point and indications of their relative flowability on beryllium T-joints.

Joints brazed with two of the alloys are shown in Fig. 4.28. It has been noted that, in general, dark areas and/or voids appear in the beryllium adjacent to the bond area in these joints, as shown in the bright-field pictures of Fig. 4.28. The nature of such areas is not definitely known, but they are believed to be intermetallics which have been formed between beryllium and the brazing alloy constituents. The voids are believed to be caused by diffusion of beryllium into the brazing alloy. Braze-metal cracks observed both visually and metallographically have indicated that many of the joints prepared thus far are brittle.

Diffusion Bonding. The study of diffusion bonding as a possible method of joining beryllium is continuing, with the use of the molybdenum fixtures described previously.²⁴ The optimum conditions for complete

²⁴"GCR Quar. Prog. Rep. Sept. 30, 1959," ORNL-2835, p. 100.

(a) BRIGHT FIELD

(b) POLARIZED LIGHT

(c) BRIGHT FIELD

(d) POLARIZED LIGHT

Fig. 4.28. Brazed Beryllium Joints. (a) and (b) show joint brazed with 49 wt % Ti-49 wt % Pb-2 wt % Be brazing alloy. (c) and (d) show joint brazed with 46 wt % Ti-46 wt % Pd-6 wt % Al-2 wt % Be brazing alloy. Excellent wetting of the base metal by the experimental brazing alloys can be seen. Unetched. 100X.

Table 4.17. Promising Experimental Brazing Alloys
for Joining Beryllium

Alloy Composition (wt %)	Flow Point (°C)	Flowability on Beryllium T-Joints
55 Pd-30 Ti-10 Al-5 Be	1150	Good
46 Pd-46 Ti-6 Al-2 Be	1100	Excellent
43 Pd-43 Ti-9.5 Al-2 Be	1150	Good
49 Pd-49 Ti-2 Be	1000	Excellent
48 Pd-48 Ti-4 Be	1000	Excellent
45 Pd-45 Ti-10 Be	1000	Excellent
80 Au-10 Ni-10 Ta	1100	Poor
48 Au-48 Ti-4 Be	1200	
70 Au-15 Ta-13 Ni-2 Be	1200	
49 Ti-49 Cu-2 Be	1100	Excellent

bonding are being determined in tests in which effects of time, temperature, and atmosphere can be correlated. Typical bonds obtained with various time-temperature combinations in both a helium atmosphere and in vacuum are shown in Fig. 4.29. Increased grain coalescence across the interface is evident with increasing temperature and time at temperature. It is also interesting to note that no appreciable grain coalescence occurs until grain growth begins. The initiation of grain growth does not occur until the bonding temperature and pressure are sufficient to cause disruption of the oxide film which appears to surround the grains of all hot-pressed beryllium. The vaporization of beryllium and/or base-metal impurities previously reported²⁵ is significantly decreased by heating in a helium atmosphere.

Fusion Welding. A fusion-welding development program is under way in an effort to develop reproducible welding techniques, establish optimum welding conditions, and evaluate the weldability of tubing made by various fabrication processes. The several welding variables, including type of current (a-c vs d-c), travel speed, and welding atmosphere

²⁵"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 142-4.

Fig. 4.29. Beryllium Diffusion Bonds Illustrating Effects of Varying the Temperature, Time, and Atmosphere Relationships. Increased grain coalescence across the interface is evident with increasing temperature and time at temperature. The use of a helium atmosphere for bonding minimized the extent of vaporization of the beryllium and/or base-metal impurities. Unetched. 250X.

(argon vs helium) are being studied as applied to the inert-gas-shielded tungsten-arc, welding process.

Examples of welds made on beryllium tubing obtained from four different vendors are shown in Fig. 4.30. Joints that were leaktight in a mass spectrometer test have been made using the edge-weld end closure design with the materials shown in Figs. 4.30b, c, d, and e. Porosity is quite pronounced in the welds on extruded tubing, as shown in Figs. 4.30a, d, and e. The penetration of the a-c weld of Fig. 4.30b is approximately one-half that of the d-c weld of Fig. 4.30c. The end caps for all these welds were machined from MTR hot-pressed beryllium moderator blocks.

The root imperfections and porosity in the end-closure welds constitute the most important joining problem. High-purity material for the end caps is now being procured as one means of remedying this condition. Other techniques to be investigated include electron-beam welding and press fitting of the end caps.

Currently, the optimum welding conditions consist of a 10-in./min weld speed, a low-current (approximately 12 amp) preheat for 3 to 4 revolutions, and a 30- to 35-amp welding current for two revolutions, followed by a slow current taper to allow postheating of the weld.

Upset Welding. A study of upset welding is under way in an effort to produce an end closure for beryllium tubing. Twenty-one upset welds have been made with the use of an INOR-8 jig and application of the British joining method previously described.²⁵ The samples presently being used consist of a 1-in.-long, 0.3-in.-i.d., 0.040-in.-wall beryllium tube with a solid beryllium end plug approximately 1/2 in. long inserted in one end. An Armco iron or mild steel deformation ring approximately 3/4 in. in outside diameter and 0.250 to 0.350 in. in thickness is placed around the tube-to-plug assembly area. The specimen and deformation ring are then placed in the INOR-8 jig between two Inconel-X dies and raised to the temperature range of 500 to 850°C by using a high-frequency induction furnace. When the sample reaches the desired testing temperature, an axial load is applied which is sufficient to cause 70 to 100% reduction of the deformation ring thickness. In

(a) NUCLEAR METALS

EXTRUDED DC

(b) BRUSH BERYLLIUM

HOT PRESSED-AC

(c) BRUSH BERYLLIUM

HOT PRESSED DC

(d) PECHINEY TUBE

EXTRUDED DC

(e) CHESTERFIELD TUBING

EXTRUDED DC

Fig. 4.30. Beryllium Tubing Fusion Welds Made Using the Machined Edge-Weld End-Closure Design. Unetched. 50X.

this temperature range, beryllium is very ductile, and deformation of the test specimen is concentrated in the area of the solid plug. Extensive radial deformation of the tube and plug toward the axis of the specimen is thus created.

Four leaktight joints have been produced. The conditions for producing a leaktight joint consisted of a 550°C initial deformation temperature and an axial pressure of approximately 3000 psi. Following deformation at 550°C, the temperature was immediately raised to 800°C for approximately 2 min to promote bonding. The sample was then air cooled to room temperature with the pressure maintained. A mild steel band was used as the deformation ring for making these specimens.

Tests conducted with initial deformation temperatures of approximately 800°C were unsuccessful. The Armco iron ring deformed easily and caused flow of the ring, tube, and plug relative to each other. Under these conditions the soft deformation ring was so plastic that random flow occurred instead of preferred-direction flow, and, since no intimate contact between the plug and tube assembly was produced, poor bonding resulted.

Metallographic examination of the leaktight joints showed a mechanical rather than a metallurgical joint. A typical joint area and a view of the over-all specimen are shown in Fig. 4.31. In the high-magnification view of Fig. 4.31b, the worked grains along the bond line and the preferred texture that exists adjacent to the boundary can be seen. These changes in the orientation and texture of the material resulted from the bonding technique.

Tube-Burst Testing of Beryllium

Procedures for fabricating suitable beryllium elevated-temperature tube-burst test specimens are being developed. An out-of-pile specimen was fabricated according to the capsule design previously described²⁵ and then tested. The capsule was pressurized to produce a hoop stress in the tube wall of 2000 psi and was tested at 750°C. Failure occurred in the tube wall in 3.2 hr. The as-fabricated specimen, the location of

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PHOTO Y 35069

Y 34319

(a) OVER ALL VIEW OF BOND AREA 6X

Y-35012

(b) CLOSE-UP VIEW OF BOND AREA

Fig. 4.31. Beryllium Tubing Upset Weld Showing Extent of Deformation and Lack of Grain Coalescence Across the Interface. Unetched.

Fig. 4.32. Photographs Illustrating Results of First Out-of-Pile, Elevated-Temperature Beryllium Tube-Burst Test. Unetched.

the failure, and photomicrographs of the area of the failure and the tube-to-end plug joint after testing are shown in Fig. 4.32. Since the end closure was made by diffusion bonding for 2 hr in a vacuum at 1100°C, a before-test view of the joint would have been similar to that shown in Fig. 4.29a. It is evident that the bond was not disturbed by the test.

5. IN-PILE TESTING OF COMPONENTS AND MATERIALS

Fuel Element Irradiation Program

Full-Diameter Prototype EGCR Fuel Capsules

ORR Experiments. Irradiation of eight full-diameter prototype group II capsules has continued, with the desired cladding temperatures being maintained for all capsules by adjustment of the helium-nitrogen thermal-barrier gas mixtures, as described in the previous report.¹ It was necessary to modify the barrier gas mixtures at the beginning of reactor cycle 24 in order to maintain design temperatures during hot-weather operation of the reactor at a power level of 16 Mw. The temperatures were about 230°F low at 16 Mw before adjustment from the gas mixtures used during operation at 20 Mw. The new mixtures contain up to 100% nitrogen. The operating temperatures recorded on June 1, 1960, and the estimated total burnup through May 21, 1960, are given in Table 5.1.

Data for the group I capsules have been analyzed. A graph showing the cladding-temperature history of capsule 01-1 is presented in Fig. 5.1. The high, low, and average temperatures for six thermocouples from a single daily reading taken about midnight are plotted against time. A similar graph with all data points recorded during reactor cycle XI of the same experiment is presented in Fig. 5.2. A plot of the reactor power and a plot of the nitrogen content of the thermal barrier gas mixture used for temperature control are also included in Fig. 5.2.

Planning and design work were completed for removal of the group II capsules at the end of reactor cycle 25. The mounting fixtures and gang-type thermal shutter devices are to be removed also and replaced with the mounting units and individual cycling devices that are being fabricated for the group III units.

The eight group III capsules that are scheduled for insertion in the ORR during August are designed for irradiation at a reactor power

¹"GCR Quar. Prog. March 31, 1960," ORNL-2929, pp. 148-50.

Fig. 5.1. Daily Cladding Temperature History of Capsule 01-1 During Irradiation in ORR.

Fig. 5.2. Detailed Cladding Temperature History of Capsule 01-1 During ORR Cycle XI.

Table 5.1. Operating Temperatures and Burnup Data for the Group II Capsules Being Irradiated in the ORR

Capsule No.	Nitrogen in Gas Mixture (%)	Capsule Temperature (°F)				Estimated Total Burnup ^a (Mwd/MT)
		Design	High	Low	Average	
01-2	40	1300 ± 50, average	1390	1036 ^b	1322	1620
02-2	80	1600 (+0,-50), high	1550	1210 ^b	1427	1630
03-2	42	1300 ± 50, average	1410	1012 ^b	1369	1840
04-2	28	1300 ± 50, average	1347	1005 ^b	1291	2090
05-2	33.5	1600 (+0,-50), high	1600	1375	1491	2040
06-2	59	1600 (+0,-50), high	1575	1440	1513	1690
07-2	46	1300 ± 50, average	1450	1235	1303	1310
08-2	100	1600 (+0,-50), high	1582	1392	1496	1130

^aBurnup is based on an accumulated reactor power generation of 60 225 Mwhr as of May 21, 1960.

^bSpecially placed thermocouples 1/4 in. from the ends of the pellet stacks in capsules 01-2 through 04-2 are indicating these low temperatures.

level of 30 Mw. These units include six beryllium-clad UO₂ capsules, two of which were provided by the United Kingdom Atomic Energy Authority and two by the French Atomic Energy Commission. The remaining two beryllium-clad UO₂ capsules are being fabricated at ORNL. One of the ORNL units is a counterpart of the 0.038-in.-o.d. British capsule and the other will be a counterpart of the 0.598-in.-o.d. French unit. Two EGCR-prototype full-diameter (0.750-in.-o.d.) stainless steel capsules containing UO₂ of different grain sizes will complete the eight group III units. Techniques are being developed for incorporation of thermocouples into these capsules for measurement of the central pellet temperature. The British capsules contain UO₂ enriched 2.45% in U²³⁵, whereas the French UO₂ is unenriched. Fabrication work is also in progress on a backup set of capsules (group III B) for use if any of the beryllium-clad units should be declared unacceptable for irradiation. The backup units consist of 0.530-in.-o.d. stainless steel cladding swaged onto UO₂ pellets.

ETR Experiments. Four capsules, designated E-1R, E-4R, E-11, and E-12 were inserted in the ETR on May 23. On the same date, three (E-3, E-8, and E-10) of the ten previously inserted assemblies were relocated to higher neutron-flux positions. This was done as a step toward irradiation of all 14 units at a revised heat rate specification of 35 000 Btu/hr·lin ft. Subsequent to the relocation and new insertion activities, 13 of the experimental assemblies were operating satisfactorily. The possibility of a plug in the gas line to capsule E-12 is indicated. Investigation of this must await the next scheduled shutdown.

Miniature Capsule Fission-Gas-Release Experiments

Irradiation of experimental assembly L-25ab was completed and irradiation of one new experimental assembly, L-28ab, was begun. Significant data and operating conditions for the experimental assemblies currently being irradiated are given in Table 5.2. Experimental assembly L-25ab was irradiated primarily to obtain thermocouple information and to observe any in-pile changes in the thermal conductivity of the UO_2 . The thermal conductivity of the UO_2 did not appear to change with irradiation at a neutron flux of 2×10^{13} neutrons/cm²·sec, as indicated by a constant central fuel temperature. Because of the nature of the fission-gas-release experiments and the reactor startup procedure, observations at times less than about 20 min are not possible. With respect to thermocouple performance, the tungsten-rhenium thermocouple in the b capsule indicated a constant temperature of ~2500°F for the first 30 days of operation; for the next 18 days, the emf output from the thermocouple steadily increased to a maximum equivalent to 2900°F on the calibration curve; then the thermocouple failed. The tungsten-rhenium thermocouple in the a capsule indicated a reasonably constant temperature of 2100°F throughout the test period.

When the thermocouples were removed, the a-capsule thermocouple appeared to be in good condition, but the entire tantalum sheath in the heated zone and a part of the thermocouple wire material of the b-capsule thermocouple had disintegrated. From these observations, it is concluded

Table 5.2. Operating Data for Miniature Capsule Fission-Gas-Release Experiments

UO₂ pellet density: 95% of theoretical

Experimental Assembly No.	UO ₂ Enrichment (% U ²³⁵)	Central UO ₂ Temperature (°F)		Date Installed
		Capsule a	Capsule b	
L-22ab	10	1380	1600	11-10-59
L-23ab	15	2800	2800	12-8-59
L-24ab	20	2600	3100	12-16-59
L-25ab	20	2100	2500	2-11-60 ^a
L-27ab	15	2000	2600	3-15-60
L-28ab	30	2200	3800	4-5-60

^aRemoved 4-5-60

that tungsten-rhenium thermocouples probably give accurate readings as long as the sheath affords protection at the junction. Upon loss of the protective sheath, however, the wire materials react with the environment and form a new junction that gives a higher output voltage for a given temperature. The L-23b-capsule thermocouple also operated at a constant temperature for 22 days, then increased in temperature from 2800°F to 3300°F over a 9-day period, and then failed. This effect was also noted for the L-27a-capsule thermocouple (initial temperature, 2000°F) and the L-24b-capsule thermocouple (initial temperature, 3100°F). The highest initial temperature (3800°F) of this series of experiments was obtained in the b capsule of experimental assembly L-28. At this high temperature, the thermocouple reading was considered to be valid for only the first 10 hr of operation, and failure occurred within 24 hr.

Advanced Fuel Element Testing

A second fueled-graphite assembly, designated ORNL-MTR-48-2, is being prepared for irradiation. A sectional view of the fuel element and its irradiation facility is shown in Fig. 5.3. The fission power density in this experiment will be 140 w/cm³, which is to be compared

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Fig. 5.3. Irradiation Facility for Second In-Pile Test of Fueled Graphite (ORNL-MTR-48-2).

with 280 w/cm³ in the first experiment, ORNL-MTR-48-1. The graphite container for the fueled matrix has a permeability of 1.5×10^{-8} cm²/sec, which is to be compared with 10⁻⁴ cm²/sec for the graphite container used in the first test. In this assembly the two UC₂-fueled graphite matrices, 1.235 and 0.233 in. long, respectively, and 0.996 in. o.d., are in a single graphite container, rather than being separated as in the first experiment.² Helium will be swept past the graphite container to carry escaping fission gases to a radiation monitor and gas-sampling station for obtaining a measurement of the fission-gas release.

Examination of Irradiated Capsules

LITR-Irradiated Miniature Capsules

Measurements have been made of the fission gas evolved by miniature capsules irradiated at maximum central UO₂ temperatures in the range 2750 to 3300°F. The data, presented in Table 5.3, indicated that, at 2900°F and above, fission-gas release increases substantially. It should be noted, however, that the oxygen-to-uranium ratios of the UO₂ pellets used in these tests were higher than the ratio of 2.00 to 2.02 specified for the UO₂ pellets of the EGCR fuel elements. The high gas evolution from capsule L-11b can be attributed to the low density, high carbon content, and high oxygen-to-uranium ratio of the UO₂. The very fine

²"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, p. 108, Fig. 5.3.

Table 5.3. Fission Gas Evolution from LITR-Irradiated Miniature Capsules

Capsule No.	UO ₂ Density (% of Theoretical)	Maximum Central UO ₂ Temperature (°F)	Burnup (Mwd/MT)	Fission Gas Evolution (%)			Oxygen-to-Uranium Ratio	Carbon Content of UO ₂ (ppm)
				Kr ⁸⁵	Xe ¹³³	Xe ¹³⁵		
L-7xa	94.99	~2750	16 000	0.074	0.16		2.0407	
L-7xb	94.85	2950	16 000	2	0.85		2.0407	
L-11b	85.23	2850	22 000	40	5.3	2.1	2.1401	630
L-10b	95.19	2900	1 600		30	27	2.0407	
L-17xa	95.49	~3000	3 100		30	4.5	2.0443	400
L-17xb	95.24	3300	3 100		61	20	2.0443	400

grain structure of the UO_2 irradiated in capsule L-10b is shown before and after irradiation in Fig. 5.4. No significant change is apparent. The grain structure of UO_2 irradiated in capsule L-17xb is shown in Fig. 5.5. Some indications of grain growth during irradiation are present.

ORR- and ETR-Irradiated Full-Diameter Capsules

Fission-gas samples from ORR-irradiated capsules O-1, O-2, O-3, and O-5 have been processed to remove the chemically active components so that mass spectrometer analyses can be made. Two samples were taken from each capsule, one being a small sample for gamma-ray spectrometer analysis and one being a large sample for mass spectrometer analysis.

The mass spectrometer sample from capsule O-1 was lost because of a leak in the sampling tank, but the small sample has been analyzed for Kr^{85} and will be reprocessed later for mass spectrometer analysis. The results of the analyses completed thus far are presented in Table 5.4.

The only quantitative analysis obtainable was that for Kr^{85} , which was measured by gamma-ray spectroscopy, since all other radioactive gases had decayed and the accurate pressure and volumetric measurements needed for converting mass spectrometer data into absolute quantities could not be obtained. Gamma-ray analyses of Kr^{85} are based on the 0.65% branching ratio. The release percentages given in Table 5.4 for the other isotopes were calculated from the Kr^{85} value and the relative abundances indicated by mass spectrometer analyses. The Kr^{85} measurement was the only one obtained for the ETR-irradiated capsule.

Postirradiation examination of ETR-irradiated capsule E-1 was completed at GEVAL. The gross-gamma-activity scan along the capsule (Fig. 5.6) indicated a very uniform neutron flux. The capsule was pierced, and an internal gas pressure at room temperature of 61.5 psia ($\pm 20\%$) was calculated from pressure measurements on two successively larger volumes. The E-1 capsule contained solid pellets, with very little free volume. Gamma-ray spectrometer analysis indicated that 5.6% ($\pm 0.5\%$) of the Kr^{85} generated was released from the UO_2 (Table 5.4).

Fig. 5.4. Structure of UO_2 in Capsule L-10b. (a) Before irradiation. (b) After irradiation. 250X.

Fig. 5.5. Structure of UO_2 in Capsule L-17xb. (a) Before irradiation. (b) After irradiation. 250X.

Table 5.4. Results of Analyses of Fission-Gas Samples from ORR- and ETR-Irradiated Full-Diameter Capsules

Capsule No.	Cladding Temperature During Irradiation (°F)	Burnup (Mwd/MT)	Type of UO ₂ Pellet	Calculated Internal UO ₂ Temperature (°F)	Fission-Gas Release (%)							
					Kr ⁸³	Kr ⁸⁴	Kr ⁸⁵	Kr ⁸⁶	Xe ¹³¹	Xe ¹³²	Xe ¹³⁴	Xe ¹³⁶
O-1	1300	1760	Hollow	2510			0.4					
O-2	1600	1910	Hollow	2820	1	0.8	0.9	0.8	0.5	0.6	0.6	C.7
O-3	1300	1640	Hollow	2670	0.4	0.3	0.5	0.4	0.2	0.3	0.3	C.3
O-5	1600	3260	Hollow	3110	0.3	0.3	0.6	0.3	0.2	0.2	0.2	C.1
E-1	1300	1000	Solid	3717			5.6					

MTR-Irradiated Fueled Graphite and Graphite Capsule

Experimental assembly ORNL-MTR-48-1, which contained two small cylinders of graphite fueled with 19 wt % enriched UC₂, was irradiated for 1400 hr in the MTR, as described previously.³ The fuel cylinders were individually canned in graphite and contained a total of 9.33 g of U²³⁵. They were irradiated at central temperatures of up to approximately 3000°F. The temperatures of the graphite walls ranged from 1100 to 1600°F during irradiation.

The fueled graphite was prepared by admixture of UO₂ spherical shot (104–147 μ) and a previously prepared carbonaceous mix. This mixture was molded to a cylindrical shape and baked. During the final baking at 2800°C, the UO₂ was converted to UC₂, and some particle enlargement occurred. An autoradiograph of the UC₂ obtained by an 18-hr exposure of contrast-process orthofilm in direct contact with the end of the fuel cylinder is shown in Fig. 5.7. The hollow spheres of UC₂ can be seen outlined in a rather uniform pattern. The autoradiograph of Fig. 5.7 was obtained prior to irradiation.

During disassembly and postirradiation examination of this experimental assembly, the gas tube protruding from the steel containment vessel was pierced, and the mixture of helium and fission gases was collected in an evacuated system. A relative gamma-activity scan, as shown in Fig. 5.8, indicated that the expected neutron flux distribution had been attained along the experimental assembly.

³"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 155–7.

Fig. 5.6. Gross-Gamma-Activity Scan of ETR-Irradiated Capsule E-1.

The two graphite containers have been removed, and visual inspection has revealed no apparent change to them or to the carbon spacers between them. These pieces are shown in Fig. 5.9. The dimensions of the graphite containers agreed with preirradiation values to within 0.1%. The dimensional data are presented in Table 5.5. The graphite-UC₂ fuel cylinder was removed intact from one of the graphite cans, and no radiation damage was apparent.

Table 5.5. Dimensional Data on Graphite Containers of Experimental Assembly ORNL-MTR-48-1

	Element A Dimensions (in.) ^a		Element B Dimensions (in.)	
	Before Irradiation	After Irradiation	Before Irradiation	After Irradiation
Average length of capsule body	2.5055	2.506	2.5006	2.501
Average diameter of capsule body	1.8753	1.875	1.8752	1.875
Average diameter of end projection				
Plug end		0.7505	0.7503	0.7510
Solid end		0.7501	0.7503	0.7507
Total length	3.2551	3.253	3.2543	3.254

^aElement A was closest to reactor center during irradiation.

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Fig. 5.7. Autoradiograph of UC₂ Dispersed in Graphite. 3X.

Fig. 5.8. Gamma-Activity Scan of Experimental Assembly ORNL-MTR-48-1.

The fueled-graphite cylinder is shown before and after irradiation in Fig. 5.10. Micrometer measurements indicated a decrease in diameter and an increase in length, as shown in Table 5.6. Precise measurements were difficult to make because the surface of the fuel cylinders are quite friable. Samples of the graphite container were submitted for qualitative radiochemical analysis. The analytical results are presented in Table 5.7 relative to Ce^{144} abundance.

Fig. 5.9. Pieces of Irradiated Assembly ORNL-MTR-48-1.

(a) BEFORE IRRADIATION

(b) AFTER IRRADIATION

Fig. 5.10. Fuel Cylinder Consisting of 19 wt % UC_2 Dispersed in Graphite. (a) Before irradiation. (b) After irradiation.

Table 5.6. Dimensional Data on Fueled-Graphite Cylinder of Element B of Experimental Assembly ORNL-MTR-48-1

	Dimensions (in.)		Change (%)
	Before Irradiation	After Irradiation	
Average diameter			
End toward reactor	0.9966	0.973	-2.4
End away from reactor	0.9966	0.946	-5.1
Average length	1.4682	1.521	+3.5

Table 5.7. Radionuclides Detected in Graphite Dust from Irradiated Experimental Assembly ORNL-MTR-48-1

Nuclides Found	Abundance Relative to Ce ¹⁴⁴	
	Observed	Expected ^a
Ce ¹⁴⁴	1	1
Ce ¹⁴¹	0.6	0.6
Zr ⁹⁵	0.08	1.6
Ba ¹⁴⁰	1.5×10^{-3}	0.02
Ru ¹⁰³	<0.02	0.4
Cs ¹³⁷	<0.02	0.04

^aBased on fissions that normally occur in U²³⁵.

ORR Closed-Cycle Loop Irradiations

Two UO₂ samples encased in siliconized silicon carbide-coated graphite supplied by Minnesota Mining and Manufacturing Company are presently being irradiated in the ORR loop at a thermal neutron flux of 7×10^{13} neutrons/cm²·sec. The general arrangement for this experiment is the same as for previous irradiation experiments in this facility.⁴

⁴"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, pp. 125-7.

The samples have been irradiated for more than 400 hr at 1800°F, and there has been no increase in the fission-product activity of the coolant. Tests are under way in an attempt to determine the leakage rate from an analysis of the gas activity. Much of the activity of the gas may be from contamination remaining in the loop after the previous experiments.

Instantaneous Fission-Gas-Release Experiment

The experimental assembly now being irradiated in the ORR instantaneous fission-gas-release experiment contains two thin plates of high-density UO_2 with thermocouples on the outside surfaces and sandwiched between them. The assembly is air cooled, and the fission power is varied by inserting or withdrawing the assembly from the reactor lattice. Helium or argon is swept over the fuel to entrain the released fission gas. This experiment was described in detail in a previous report.⁵

The measurement of fission-gas evolution from the UO_2 is complicated by two factors. First, the daughter products of xenon and krypton adhere to the walls of the counting system and produce a variable background. This is measured by purging the system with pure helium so that only the nongaseous fission products remain. Second, the iodine released from the fuel plates onto the walls of the sweep system, where it decays to xenon. In this manner some xenon is produced that emerged from the fuel as iodine. This contribution to the total xenon evolution can be measured only by withdrawing the fuel from the reactor to allow the temperature to decrease to the point where diffusion of gas in the fuel is essentially zero and then measuring the continued xenon production of the system.

The rate of iodine production in the fuel is entirely a function of the neutron flux, whereas the rate of iodine release, at equilibrium, is a function of both the flux and the fuel temperature. At a constant temperature and constant flux the rate of iodine release should be constant, and the iodine should plate onto the sweep-gas system until the rate of decay equals the rate of iodine release.

⁵"GCR Semiann. Prog. Rep. June 30, 1959," ORNL-2767, pp. 164-9.

Because of the effect of the second factor on the total gas production of the fuel, a study was made of the temperature dependence of iodine release from the fuel. For this study, the flux was maintained constant and the fuel temperature was set at an arbitrary level. The fuel was withdrawn from the reactor after various lengths of time under constant conditions, and the xenon activity was measured. The xenon-activity data obtained in this manner are plotted in Fig. 5.11 as a function of the temperature of the fuel when the iodine was released (before withdrawal). The activity of the xenon gas that evolved from the fuel, rather than being produced from the plated iodine, is also plotted in the same manner for

Fig. 5.11. Xenon Evolved from UO₂ Fuel and Produced from Iodine Released from Fuel in Instantaneous Fission-Gas-Release Experiment.

comparison. The iodine release appears to have the same temperature dependence as that of the release of xenon and krypton.

The numbers by the data points in Fig. 5.11 give the hours at steady-state conditions before the fuel was withdrawn. These data demonstrate that only about 2 hr is required to obtain an equilibrium production of xenon from the plated iodine. This rapid equilibrium of the xenon is an unexplained phenomenon. When the xenon production is constant, the plated iodine has a decay rate equal to the rate of deposition, and for uniform evolution of iodine from the fuel, this requires around five half-lives of the iodine. The greater proportion of the xenon activity is initially due to Xe^{135} and, since its parent, I^{135} , has a 6.7-hr half-life, this should require more than 30 hr for equilibrium.

The total escape of xenon and krypton from the fuel, regardless of method of release, is of primary importance. In order to determine the rate of gas release as a function of temperature, the flux was held constant and the fuel temperature was set at an arbitrary level. When the gas activity had reached equilibrium, samples of sweep gas were trapped and analyzed for Xe^{135} and Kr^{88} . The samples were taken over a period of several days at random temperatures. The results of these samplings are given in Fig. 5.12. The numbers by the data points give the sequence of measurement and demonstrate that the lines represent equilibrium values. One measurement was made at a higher flux but an equal temperature. The amount of gas released was greater at the higher flux, as would be expected. The flux was measured by argon activation when argon was used as the sweep gas.

The samples of gas marked A and B on Fig. 5.12 were analyzed six months prior to the current samples, and the data are included to show that the escape rate was lower at that time, even though the temperature and flux were higher. The flux was determined by thermal measurements for samples A and B. The fractional release of gas from the fuel has not only increased by more than a factor of 100 since samples A and B were measured, but the relative proportion of xenon and krypton has changed so that a greater proportion of xenon is now released (see Table 5.8).

Fig. 5.12. Fission Gas Released from UO_2 as a Function of Fuel Temperature at Constant Flux.

The fractional release obtained for samples A and B agrees with that reported for capsule puncture tests.^{6,7}

Diffusion coefficients were calculated in the manner described in the previous report,⁸ using a slab model. The diffusion coefficients were not calculated for Xe^{135} , since it is apparent that a large fraction of the Xe^{135} escapes from the fuel as I^{135} . The parent of Kr^{88} is Br^{88} , which has a 15-sec half-life. This is short enough to neglect Br^{88} diffusion. The diffusion coefficients, Fig. 5.13, show a reasonable

⁶"GCR Quar. Prog. Rep. Sept. 30, 1959," ORNL-2835, p. 117.

⁷"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, p. 164.

⁸Ibid., pp. 173-8.

Table 5.8. Release of Kr^{88} and Xe^{135} During Steady-State Operation of Instantaneous Fission-Gas-Release Experiment in ORR

Sample	Date of Sampling	Flux (neutrons/cm ² ·sec)	Fuel Temperature (°F)	Fission Gas Release (%)	
				Kr^{88}	Xe^{135}
A	October 1959	5×10^{13}	1945	0.031	0.036
B	October 1959	5×10^{13}	1890	0.017	0.018
1	April 6, 1960	3.3×10^{12}	1220	0.3	1.1
2	April 6, 1960	3.3×10^{12}	1380	0.6	1.4
3	April 6, 1960	3.3×10^{12}	1570	1.5	3.2
4	April 8, 1960	3.3×10^{12}	1640	1.2	2.5
5	April 8, 1960	3.3×10^{12}	1760	2.2	5.7
6	May 3, 1960	3.3×10^{12}	1500	1.3	4.0
7	May 4, 1960	3.3×10^{12}	1500	1.2	2.9
8	May 6, 1960	5.9×10^{12}	1700	1.6	6.1

Fig. 5.13. Calculated Diffusion Coefficient Values for Kr^{88} Based on Data Obtained in Instantaneous Fission-Gas-Release Experiment. Numbers give time order of measurements.

temperature dependence but are more than four orders of magnitude larger than those measured for samples A and B.

When the temperature of irradiated UO_2 is increased, a burst of activity is released. This burst implies that a store of fission gas is released when the temperature is increased. These bursts may peak at twice the equilibrium activity, and equilibrium is subsequently attained within 2 hr. One massive burst of activity was obtained when the fuel temperature was increased by substituting argon for helium as a sweep gas (the lower thermal conductivity of argon caused a 400°F increase in the fuel temperature). This sweep gas substitution normally results in a burst of activity because of the temperature increase, but the magnitude of this burst was without precedent. As shown in Fig. 5.14, where the activities are plotted for the time period just before the burst and for the burst, the activity of the burst was more than six times that expected for the temperature and flux. This burst caused the activity alarm system for the experiment to trip so that the fuel automatically withdrew from the reactor and the sweep gas system was isolated. The isolation valves were then opened for a moment to sample some of the gas. The spectrum of the gas was unusual and was dominated by Xe^{133} , Xe^{135} , Kr^{88} , Kr^{87} , and $\text{Kr}^{85\text{m}}$, all long-lived gases. The total amount of gas released could not be calculated because it was necessary to valve off the system to prevent the burst activity from passing outside the reactor shielding.

The magnitude of this burst, which occurred during routine operation, demonstrates a rather abrupt recent change in the fission-gas retention characteristics of the fuel. The increased gas release rate, compared with that measured six months before, shows that a previous change in retention characteristics had occurred. Recently, during a routine insertion of the fuel into the reactor, a temperature excursion was observed that could only be explained as the release of stored energy. The conditions for storing energy in fuel were repeated (neutron irradiation at low temperatures), and a stored energy release was again observed. The energy release began in the range 500 to 600°F , and 4 to 6 cal/g of

Fig. 5.14. Fission-Product Activity Prior to and Following Activity Burst in Instantaneous Fission-Gas-Release Experiment.

energy was released. Stored energy has never been observed in UO_2 with an oxygen-to-uranium ratio of 2.00, but stored energies ranging up to 23 cal/g have been reported⁹ for UO_2 with an oxygen-to-uranium ratio of 2.08 or greater.

The observation of stored energy in the fuel indicates that the oxygen-to-uranium ratio of the fuel (initially 2.01) has increased to 2.08 or more. It seems more likely that the increased fission-gas release, as well as the large bursts, are the results of oxidation of fuel rather than irradiation damage. The sweep gases used are passed through purification systems prior to use, but the efficiency of the removal of trace quantities of oxygen is doubtful. Even 1 ppm O_2 in the gas stream could appreciably change the oxygen-to-uranium ratio over a period of

⁹B. C. Childs and J. McGum, "The Differential Calorimetry of Irradiated UO_2 and U_4O_9 ," GRMet-893 (December 1959).

months. An improved gas purification system will be installed for the next run.

Radiation Effects on Structural Metals

Type 304 Stainless Steel Tubing

Type 304 stainless steel of the type to be used as a cladding material for the EGCR fuel elements is being tested in in-pile tube-burst-type stress-rupture tests. A few specimens were tested some time ago, and, when the results were compared with out-of-pile data, no irradiation-induced change in time to rupture was found for tests of up to 1000 hr duration. Recent tests of samples from the same heat of material (Superior Tube Company, heat No. 23999X) have indicated, however, that the time to rupture is reduced by a factor of 2 when the material is subjected to neutron bombardment at 1500°F. Seven of the ten tube-burst specimens of the same heat now being tested in the poolside facility of the ORR at 1300°F in air with the reactor at a power level of 16 Mw have ruptured. The data obtained thus far are summarized in Table 5.9. Out-of-pile tests under similar conditions will be started soon on specimens from the same heat of material.

Heavy Steel Plate

Irradiation test specimens are being fabricated from five, heavy, steel-plate weldments made with E-7016 electrodes. The weldments were inspected by both ultrasonic and x-ray techniques and were found to be satisfactory. The steel plates were selected from materials considered for the fabrication of reactor pressure vessels. Apparatus for irradiation of these specimens in the ORR poolside facility at elevated temperatures under various neutron flux conditions is being designed.

Studies of irradiation effects in steels have, in most instances, involved exposures of several months duration at high neutron fluxes. Extrapolation of data from such studies to obtain engineering design information for 20-year service at low neutron fluxes is not presently

Table 5.9. Results of Tube-Burst Tests
of Type 304 Stainless Steel at 1300°F
in Air Under Irradiation in the
ORR Pool-Side Facility

Specimen No.	Stress (psi)	Time to Rupture (hr)
15-2	13 000	130
15-3	13 000	197 ^a
15-6	13 000	144
15-8	13 000	122
15-4	11 000	512
15-7	11 000	480
15-10	11 000	462
15-1	9 000	>925 ^b
15-5	9 000	>925 ^b
15-9	9 000	>925 ^b

^aThis specimen received 8 303 Mwhr of
neutron bombardment at 1300°F prior to
application of the stress.

^bTest continuing.

considered to be reliable. Therefore plans are being made to obtain information on the effects of long-term exposures. The first study will involve exposure of test specimens at various controlled temperatures in the ORNL Graphite Reactor for periods of up to two years. The specimens will be fabricated from the same steels as those used for the shorter-period irradiations in the ORR at higher neutron fluxes. The design of the facilities for the Graphite Reactor irradiations is nearing completion.

The second long-term study will consist of surveillance tests of specimens exposed in operating power reactors for periods of up to 20 years. These specimens will be fabricated of the material used in the construction of the reactor. Exposure temperatures will not be artificially controlled, but they will approximate the temperature of the corresponding reactor component as closely as is practicable. The specimens will be placed so as to be exposed to about the same neutron spectrum and flux as the reactor component or to a slightly higher neutron flux. The results

of these tests will give information relative to the actual condition of the components of the particular reactor and data on long-term irradiation effects. The long-term data will give a basis for the extrapolation of results from short-term tests. Such surveillance specimens will be included in the N.S. Savannah reactor and in the EGCR.

Beryllium

The first high-temperature beryllium irradiation experiment¹⁰ was completed in the ORR, and postirradiation examination of the specimens was initiated. Although it was originally planned to terminate the experiment after a fast-neutron (>1 Mev) exposure of 10^{21} neutrons/cm², technical difficulties necessitated early removal of the experimental assembly. The fast-neutron exposure was 7×10^{19} neutrons/cm².

Failure of the furnace circuits, probably because of hydrogen embrittlement of the copper lead wires, was responsible for the early termination of the experiment. The circuit failures resulted in loss of close control of the specimen temperatures, although, even without the heaters, the specimen temperatures remained in the 500 to 600°C temperature range during the experiment.

Postirradiation density measurements have been completed, and the results are presented in Table 5.10. The density changes indicate that swelling of the coupons occurred. The temperatures indicated were measured by Chromel-Alumel thermocouples and varied somewhat during the experiment. The flux measurements were made with the use of neptunium monitors. The limited number of measurements that have been made in the B-8 lattice position of the ORR have defined the integrated flux to roughly $\pm 25\%$. Further measurements will increase the precision of the dose levels. An indication of the spectral distribution in the B-8 position is being obtained with the use of sulfur, cobalt, and U²³⁸ monitors.

The data indicate density decreases of 1.5 to 5% during irradiation. There appears to be no correlation with temperature in the 500 to 600°C

¹⁰"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 179-84.

Table 5.10. Data Showing Effects of Irradiation in ORR for 24 Days on Density of Beryllium Specimens Prepared by Various Techniques

Type of Material From Which Specimen Was Machined	Heat Treatment Prior to Irradiation	Irradiation Temperature (°C)	Integrated Flux at Energies Above 0.66 Mev (neutrons/cm ² , ±25%)	Density Change (%, ±0.5)
			×10 ²⁰	
Hot-pressed block	Annealed at 800°C for 2 hr	615	1.0	-1.9
	Annealed at 800°C for 2 hr	535	1.3	-2.3
	Annealed at 1100°C for 2 hr	560	1.3	-1.9
Unidirectionally rolled sheet	Annealed at 800°C for 2 hr	600	1.0	-2.8
	Annealed at 800°C for 2 hr	520	1.3	-4.9
	Annealed at 800°C for 2 hr	490	1.0	-2.7
	Annealed at 800°C for 2 hr	560	1.3	-2.9
	Annealed at 800°C for 2 hr	510	1.3	-2.7
Rod	Annealed at 800°C for 2 hr	550	1.2	-4.7
Cross-rolled sheet	Annealed at 800°C for 2 hr	615	1.2	-2.6
	Annealed at 800°C for 2 hr	515	1.3	-1.4
	Annealed at 800°C for 2 hr	530	1.2	-2.7
	Annealed at 800°C for 2 hr	575	1.2	-1.6
	Annealed at 800°C for 2 hr	605	0.7	-2.7
	Annealed at 800°C for 2 hr	505	1.3	-3.6
	Annealed at 1100°C for 2 hr	590	1.3	-3.3

range of experiment. The neutron dose levels are not yet known to sufficient accuracy to expect a correlation with the density changes. A trend may be seen, however, in the relationship between the fabrication history and the density change. The average density decrease for the specimens machined from hot-pressed block is less than for the wrought specimens, that is, the rod specimens and the unidirectionally and cross-rolled sheet specimens. This difference may be explained by the fact that the preirradiation density of the block material was lower than that of the wrought material, implying that densification was not completed during hot pressing. The voids created in the fabrication process may have closed before the apparent density (as measured by immersion) started to decrease as the result of the accumulation of helium and the formation

of bubbles within the beryllium. The British¹¹ have observed a similar effect when comparing the data obtained during postirradiation annealing of hot-pressed and wrought beryllium specimens.

The data of Ells¹² on postirradiation annealing of MTR shim-rod beryllium irradiated at low temperatures indicate a density decrease of 1.5% in 24 days at 595°C. This beryllium had received a fast-neutron (>1 Mev) dose of 10^{22} neutrons/cm², which is roughly a factor of 100 greater exposure than that of the ORNL specimens irradiated at high temperatures. The lack of correlation between the data from the two types of experiments is surprising in view of the previous experience on the relationship between swelling of uranium alloys on postirradiation heat treatment and on hot irradiation. Speculation as to the reasons for the apparently anomalous behavior is not warranted until the helium content of the specimens has been determined and the metallographic examinations have been completed. In addition, an out-of-pile mockup of the experiment will be necessary to reproduce the environmental history of the specimens so that any error inherent in the system may be detected.

As a result of the difficulty with the copper lead wires to the furnace in the first irradiation experiment, the He-H₂ mixture will be discarded in favor of a helium atmosphere. The copper wire will be replaced with electrolytic nickel wire in the high-flux, high-temperature region, and, in order to reduce the mass of the experiment, the beryllium specimens will not be encapsulated in stainless steel. Otherwise, the second experimental assembly will be similar to the first, which was described previously.¹⁰

¹¹Private communication from J. B. Rich, Harwell, May 16, 1960, to J. R. Weir, ORNL.

¹²C. E. Ells, "The Swelling of Beryllium from Neutron Induced Gases," CRMet-809 (November 1958).

Inconel

The data obtained during the quarter from in-pile tube-burst tests of Inconel are summarized in Table 5.11 and may be compared with the data presented previously.¹³ Work continued on the fabrication of specimens from special boron-controlled heats. The out-of-pile tests of specimens from these special heats have been started.

¹³"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 184-6.

Table 5.11. Effect of Neutron Bombardment in
ORR Poolside Facility on Time to Rupture of
Inconel (CX-900 Quality) at 1500°F in Air

Specimen No.	Stress (psi)	Time to Rupture (hr)
14-6	4000	181
14-9	4000	108
14-4	3000	443
14-7	3000	441
14-8	3000	299

6. OUT-OF-PILE TESTING OF MATERIALS AND COMPONENTS

Compatibility Tests of Graphite, Structural Materials, and Helium

Polythermal Tests with Static Helium

Isothermal pot tests of graphite and structural materials in static helium have demonstrated the importance of metal-gas reactions in controlling the graphite-induced buildup of impurities in helium.¹ Thus both the area and temperature structure of metal surfaces become important factors in projecting the results of compatibility experiments as a means of predicting conditions in the EGCR. A series of polythermal tests has therefore been carried out in which the temperatures and surface areas of metal relative to graphite were varied. In these tests, tubular specimens of type 304 stainless steel, corresponding to fuel element cladding, have been held at temperatures ranging from 1500 to 1800°F while the temperature of the graphite and metal surroundings have been maintained at 1100°F.

As discussed previously,² two general test arrangements were utilized for the experiments. In one arrangement the ratio of the area of metal surface held at 1100°F to the area of the heated element was 250:1, and in the other arrangement it was 53:1. The ratio of the volume of gas to the volume of graphite was 4.9:1 in the first type of arrangement and 3.6:1 in the second type of arrangement.

The helium impurity concentrations attained in these tests and the reaction films produced on the heated elements differed markedly in the two arrangements. As may be seen in Table 6.1, the initial concentrations of CO were of the same order of magnitude in all tests; however, the CO levels declined much earlier and faster in the high surface-area ratio tests than in the low ratio tests. As shown in Table 6.2, similar changes

¹"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 187-91.

²"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, pp. 141-5.

Table 6.1. CO + N₂ Concentrations Found in Polythermal Tests of AGOT-Grade Graphite and Type 304 Stainless Steel Heated Elements in Static Helium

Test pot and graphite held at 1100°F

Metal-Surface-Area Ratio*	Heated Element Temperature (°F)	CO + N ₂ Concentration in Helium (vol %)			
		Initially	After 10 hr	After 100 hr	After 900 hr
250:1	1500	0.697	0.407	0.123	0.047
	1600	0.465	0.224	0.0479	0.041
	1700	0.632	0.577	0.072	0.048
	1800	0.612	0.485	0.205	0.047
53:1	1500	0.435	0.101	0.181	0.998
	1600	0.341	0.110	0.566	0.418
	1700	0.368	0.159	1.02	0.328
	1800	0.825	1.59	1.36	0.614

*Ratio of area of metal surface held at 1100°F to surface area of heated element.

Table 6.2. CO₂ Concentrations Found in Polythermal Tests of AGOT-Grade Graphite and Type 304 Stainless Steel Heated Elements in Static Helium

Test pot and graphite held at 1100°F

Metal-Surface-Area Ratio*	Heated Element Temperature (°F)	CO ₂ Concentration in Helium (vol %)			
		Initially	After 10 hr	After 100 hr	After 900 hr
250:1	1500	0.070	0.050	0.015	0.002
	1600	0.045	0.024	0.001	0.000
	1700	0.091	0.044	0.008	0.002
	1800	0.031	0.042	0.017	0.005
53:1	1500	0.083	0.087	0.091	0.086
	1600	0.107	0.076	0.142	0.069
	1700	0.070	0.064	0.065	0.052
	1800	0.191	0.120	0.124	0.059

*Ratio of area of metal surface held at 1100°F to surface area of heated element.

in the CO₂ concentrations were found in the two types of tests. The CO₂ levels reached their maximum in the first few hours at temperature in both types of tests and then tended to remain constant or decline very slowly in the low metal-surface-area ratio tests. In contrast, the CO₂ concentrations declined sharply in the high metal-surface-area ratio tests.

The concentrations of non-oxygen-bearing impurities were roughly comparable in both types of tests. The H₂ and CH₄ concentrations, which are shown in Tables 6.3 and 6.4, respectively, reached their maximum levels by 100 hr and usually decreased steadily thereafter. The ratios of CH₄ to H₂ were relatively large and favored carburization of the alloys under test at all time periods.

The differences in impurity concentrations in the two types of tests were, as would be expected, manifested in differences in the microstructures and surfaces of the type 304 stainless steel heated elements. The surface films on the tubes exposed in the high metal-surface-area ratio tests were silvery in color and were found by electron diffraction analysis to be composed of Fe₃O₄ and Cr₂O₃. The surface films on the tubes from

Table 6.3. Hydrogen Concentrations Found in Polythermal Tests of AGOT-Grade Graphite and Type 304 Stainless Steel Heated Elements in Static Helium
Test pot and graphite held at 1100°F

Metal-Surface-Area Ratio*	Heated Element Temperature (°F)	H ₂ Concentration in Helium (vol %)			
		Initially	After 10 hr	After 100 hr	After 900 hr
250:1	1500	1.78	2.30	1.73	0.068
	1600	2.12	2.16	1.89	0.053
	1700	2.78	3.68	5.47	0.808
	1800	2.61	2.93	3.11	0.294
53:1	1500	1.49	1.82	2.12	1.25
	1600	1.86	2.36	1.17	0.275
	1700	1.40	2.23	2.00	0.085
	1800	4.11	3.62	2.46	0.158

*Ratio of area of metal surface held at 1100°F to surface area of heated element.

Table 6.4. CH₄ Concentrations Found in Polythermal Tests
of AGOT-Grade Graphite and Type 304 Stainless Steel
Heated Elements in Static Helium

Test pot and graphite held at 1100°F

Metal-Surface-Area Ratio*	Heated Element Temperature (°F)	CH ₄ Concentration in Helium (vol %)			
		Initially	After 10 hr	After 100 hr	After 900 hr
250:1	1500	0.949	1.71	1.10	0.000
	1600	1.25	1.16	0.854	0.000
	1700	1.62	1.97	0.006	0.000
	1800	0.916	0.797	0.304	0.000
53:1	1500	0.570	0.732	0.789	0.402
	1600	0.746	0.953	1.98	0.058
	1700	0.735	0.852	0.441	0.000
	1800	1.42	0.623	0.033	0.000

*Ratio of area of metal surface held at 1100°F to surface area of heated element.

the low metal-surface-area ratio test, on the other hand, were dark green to black in color and were found to be primarily 2Cr₂O₃·Fe₂O₃, which was covered in some cases with a layer of graphite. More detailed results of the film analyses are presented in Table 6.5.

The heated elements used in both types of tests showed increases in carbon content compared with the "as-received" level of 0.02 wt %. The carbon percentages determined after the tests with the reaction films intact are shown in Table 6.5. As may be seen the amount of carbon pickup increased with increases in element temperatures. It may also be noted that the greater carbon increases occurred in the specimens used in the arrangements having the higher metal-surface-area ratio. Metallographic examinations of these specimens showed measurable increases in carbide precipitation in all cases with carbon contents of above approximately 0.1 wt %.

The interplay between graphite-gas and metal-gas reactions in the arrangements having the higher metal-surface-area ratio resulted in an environment that, throughout most of the test period, was carburizing

Table 6.5. Results of Analyses of Surface Films Formed on Type 304 Stainless Steel Heated Elements Exposed for 900 hr in Static Helium in a Polythermal System Containing AGOT-Grade Graphite Maintained at 1100°F

Metal-Surface-Area Ratio ^a	Heated Element Temperature (°F)	Appearance and Analysis of Surface Film	Carbon Content of Heated Element ^b (wt %)	Metallographic Appearance of Heated Element
250:1	1500	Silver colored; Fe ₃ O ₄ , Cr ₂ O ₃	0.17	Light carbide precipitation; very small amount of sigma phase
53:1	1500	Dark green ^c	0.03	No noticeable change in microstructure
250:1	1600	Silver colored; Cr ₂ O ₃ , Fe ₃ O ₄ , traces of 2Cr ₂ O ₃ ·Fe ₂ O ₃	0.69	Heavy carbide precipitation; small amount of sigma phase
53:1	1600	Dark green ^c	0.08	No noticeable change in microstructure
250:1	1700	Silver colored; Cr ₂ O ₃ , Fe ₃ O ₄ , Cr ₂₃ C ₆	0.59	Heavy carbide precipitation; almost continuous network of sigma phase
53:1	1700	Dark green to black; graphite layer on surface; 2Cr ₂ O ₃ ·Fe ₂ O ₃ , MnO ₂	0.11	Light carbide precipitation; almost continuous network of sigma phase
250:1	1800	Silver colored ^c		
53:1	1800	Dark green to black; graphite layer on surface; Cr ₂ O ₃ , Cr ₂ O ₃ ·Fe ₂ O ₃	0.14	No noticeable carbide precipitation; almost continuous network of sigma phase

^aRatio of area of metal surface held at 1100°F to surface area of heated element.

^bCarbon content of as-received type 304 stainless steel was 0.02 wt %.

^cAnalytical results not yet available.

and only weakly oxidizing with respect to the heated elements. Thus the relatively large increases in the carbon content of the test specimens are understandable, since relatively clean metal surfaces were exposed to the carburizing atmospheres at temperatures at which the diffusion rate of carbon is rapid. The increase in carbon content with element

temperature was probably due to the increase in the carbon diffusion rate with temperature. The oxide films that were formed during these tests were composed chiefly of Fe_3O_4 , which has characteristically been found in tests in atmospheres containing a high percentage of CO relative to CO_2 .

The atmospheres of the lower metal-surface-area ratio tests were initially carburizing to the heated elements and, in contrast to the higher surface-area ratio tests, were strongly oxidizing. The formation of a tightly adherent oxide film early in these tests could be expected to hamper the diffusion of carbon, and such films probably account for the generally lower carburization rates. The fact that a carburizing potential existed in these tests was evidenced by the deposition of graphite on the oxide surface. It may be noted that no Fe_3O_4 was detected in the films formed on the type 304 stainless steel heated elements in these tests, but, rather, the films consisted of the spinel $2\text{Cr}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$, MnO_2 , and Cr_2O_3 .

These tests clearly indicate that in a reactor system such as the EGCR the area of metal surface exposed at temperatures approximating the exit gas temperature will have an important bearing on the corrosion behavior of the somewhat hotter fuel cladding. Increasing the amount of metal held at 1100°F in these tests served to gather oxygen-containing gas contaminants at a faster rate and thereby considerably reduce the oxidation of the hotter surfaces presented by the heated element. Accordingly, the carburization rate of the heated elements was faster in the absence of an oxide film. In these test series, the arrangements having the metal-surface-area ratio of 53:1 approximated the conditions of the EGCR.

The metallographic evidence of sigma-phase material in the heated specimens is disturbing. Special etching techniques have given positive indications of sigma formation in most of the type 304 stainless steel heated elements tested to date. As noted in Table 6.5, this effect has been especially evident in elements tested at 1700 and 1800°F . Nearly continuous networks of sigma phase were formed along grain boundaries. No apparent correlation was found between the occurrence of this phase

and the gaseous environment or the subsequent differences in carbon content of the elements. The sigma phase was preferentially oxidized by the test environments, and, at the higher temperatures, intergranular attack to a maximum depth of approximately 3 mils resulted.

Low-Pressure Thermal-Convection Loop Tests with Helium Containing Controlled Amounts of Impurities

Two low-pressure thermal-convection loop tests have now been completed in which the impurity content of the circulating helium was maintained continuously in the range of EGCR specifications.³ These two tests, test No. 10, which was described previously,⁴ and test No. 11, were run at a hot-leg temperature of 1100°F. The loops contained, in addition to the helium atmosphere, graphite and specimens of structural materials. In order to obtain the desired impurities in the helium atmosphere, the graphite was allowed to outgas slowly and the system purged with helium until an effectively pure atmosphere was attained. Measured amounts of the specific impurity to be tested were then admitted to establish a fixed impurity concentration. These levels, with the exception of the H₂O content, were measured with an on-stream gas chromatograph that automatically sampled the gas stream at 20-min intervals. The H₂O content was measured by means of a "moisture monitor" and dew-point indicator.

The helium atmosphere used in test No. 10 contained 0.185 vol % CO, as a result of the injection of a measured amount of pure carbon monoxide into the loop. As shown in Table 6.6, the helium was essentially free of all other impurities, except for 0.132 vol % CO₂, which was present by virtue of the CO-graphite equilibrium.

Test No. 11 was operated with helium containing H₂O controlled to a level of approximately 0.14 vol %. Frequent additions of H₂O-saturated helium were required to maintain this level, and thus there were some fluctuations of H₂O concentration. However, the periods of fluctuation

³"EGCR Preliminary Hazards Summary Report," ORO-196 (May 1959).

⁴"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, p. 195.

Table 6.6. Maximum Impurity Concentrations in Controlled-Environment Low-Pressure Thermal-Convection Loops

	Impurity Concentration in Helium (vol %)					
	CO ₂	CO	O ₂	CH ₄	H ₂ O	H ₂
Test No. 10	0.132	0.185	0.0010	0.0012	0.0020	0.0090
Test No. 11	0.040	0.060	0.0009	0.010	0.14	0.16
EGCR helium specification	0.2				0.1	0.1

were relatively short (a few hours). The maximum concentrations of all impurities are listed in Table 6.6.

The extent of oxidation of the low-alloy structural materials in the loops is indicated in Table 6.7. Since the films on the T-1 steel specimens spalled severely, the weight change data were based on the weight loss measured after removal of the entire film. In the case of the Croloy 2 1/4 and Croloy 3M specimens, the films remained intact, and therefore the weight data were based on the weight gained during test. In both cases, the subsequent calculations of metal loss were based on the assumption that the films were entirely Fe₃O₄.

Some indication of the behavior of AISI 502 steel in the EGCR environment can be obtained by comparing the data obtained in these tests with published data on the oxidation of steels.^{5,6} In the thermal-convection loop tests the resistance to scaling of the Croloy 2 1/4 and Croloy 3M specimens was only slightly better than that of the T-1 steel specimens. Representative published data for plain carbon and low-chromium-content steels tested in air at 1100°F are compared in Table 6.7 with the data obtained in tests 10 and 11. The data indicate that additions of 2.25 to 3% chromium reduce weight gains in 1000-hr tests

⁵H. H. Uhlig, "The Corrosion Handbook," pp. 644-7, Wiley, 1948.

⁶"Steels for Elevated Temperature Service," United States Steel Company, 1949, pp. 21-23.

Table 6.7. Data Showing Effect of Environment on Low-Alloy Steels
Tested for 1000 hr at 1100°F

Data Source	Alloy	Weight Gain (mg/cm ²)	Weight of Film (mg/cm ²)	Weight of Metal That Reacted (mg/cm ²)
Test No. 10 with helium containing CO	T-1 steel	3.9	14.1	10.2
	Croloy 3M steel (3% Cr, 1% Mo)	2.4	8.6	6.2
Test No. 11 with helium containing H ₂ O	T-1 steel	7.4	26.7	19.3
	Croloy 2 1/4 steel (2.25% Cr, 1% Mo)	4.4	15.8	11.4
Published results for steels oxidized in air	Carbon steel	27	98	71
	Steel containing 2.25% Cr and 1% Mo	15	54	39
	Steel containing 5% Cr and 0.5% Mo	5	18	13

by a factor of approximately 2. Increasing the chromium content to 5% (comparable to type 502 steel) further decreased the oxidation rate in air to a factor of approximately 5 below that for plain carbon steel. Unfortunately, these data do not make allowance for scaling upon thermal cycling. The literature places the upper temperature limit for acceptable scaling resistance for the 5%-Cr steel at 1150°F, and the maximum recommended temperature for 2.25%-Cr steel in air is below 1100°F.

High-Pressure Thermal-Convection Loop Tests

Two additional tests (Nos. 6 and 7) in high-pressure thermal-convection loops containing helium, AGOT-grade graphite, metal test specimens, and a dummy fuel element assembly have been completed. The tests were each of 1000 hr duration; the system pressure was 320 psig; and the hot-leg temperatures were 1400°F in test 6 and 1100°F in test 7.

Gas analyses showed the maximum impurity concentrations in the helium used in the tests to be 3.90 vol % in test 6 and 1.15 vol % in

test 7. The maximum values were recorded shortly after reaching the test temperature, and, as in previous tests,^{7,8,9} the impurity concentration decreased during the remainder of the test period to concentrations of 1.90 vol % in test 6 and 0.62 vol % in test 7. In test 6, the final impurities were primarily $N_2 + CO$, whereas in test 7 the final impurity was primarily CH_4 .

Visual examination of specimens from tests 6 and 7 showed the stainless steels to be covered with tightly adherent, iridescent surface films. The low-alloy steels had loosely adherent, silvery gray films that spalled when the samples were removed from the loop. The dummy fuel element assembly in test 6 contained four rods loaded with UO_2 pellets. Visual examination of the assembly showed that the tubes had collapsed around the UO_2 pellets and that bowing of individual rods had occurred. Examinations of the cold legs of both loops for carbon mass transfer showed no detectable deposits.

Gas-Purification System Tests

A small-scale gas-purification system of the type proposed for use with the EGCR¹⁰ coolant system has been installed on the forced-circulation loop, and preliminary tests for determining the effectiveness of the system in removing various gaseous impurities from the circulating helium have been initiated.

The purification system includes a catalytic converter containing Girdler type G-43 catalyst for converting H_2 and CO to H_2O and CO_2 , a cooler-condenser and a silica-gel adsorber for removal of H_2O , and a molecular-sieve adsorber for removing CO_2 . The effectiveness of the system is measured with an on-stream Greenbrier gas chromatograph and a Consolidated Electrodynamics moisture monitor. Gas samples are fed to

⁷"GCR Quar. Prog. Rep. Sept. 30, 1959," ORNL-2835, pp. 125-32.

⁸"GCR Quar. Prog. Rep. Dec. 31, 1959," ORNL-2888, pp. 151-2.

⁹"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 190-3.

¹⁰F. A. Anderson, "Proposed Helium Purification System for the Experimental Gas-Cooled Reactor (EGCR)," ORNL-2819 (Oct. 2, 1959).

these instruments from various locations within the purification system and from the main test loop.

Two tests have been conducted in order to establish criteria for injection of the oxygen required for operation of the catalytic converter. During these tests the main loop was operated at approximately 100°F, and the purification system preheater was used to heat the bypass gas stream to 1050°F before it entered the catalytic converter. In the first test, the H₂ concentration in the main-loop helium stream was raised to 0.13% and a flow of 2.1 lb/hr was established through the purification system. Oxygen was then injected at the inlet of the catalytic converter at the rate of 10 cm³ (STP)/min. After 32 min, O₂ was observed at the converter exit, and O₂ injection was stopped. Circulation of the loop gas through the purification system was continued, and approximately 30 min later all H₂ had been removed from the loop gas. Excess oxygen was observed at a level of 8 ppm.

The hydrogen concentration of the main loop helium stream was then raised to 0.74% and, with the same flow rate through the purification system, O₂ was injected at the rate of 5 cm³ (STP)/min. The H₂ concentration in the loop was reduced from 0.74% to 0.02% within 5 1/2 hr, while maintaining a moisture content of less than 10 ppm. Difficulties encountered with the gas chromatograph at this point interfered with completion of the test. Additional tests with lower purification system flow rates are in progress.

Evolution of Gases from Graphite

Experimental studies of the degassing behavior of various types of graphite have been continued with the use of the techniques described previously. Experiments with Great Lakes Corporation R-6HLM (R-2) graphite have been completed, and several graphites prepared from needle coke by the Great Lakes Corporation, the National Carbon Company, and the Speer Carbon Company have been degassed at various temperatures. Some special samples, including a highly purified AGOT-grade graphite,

a low-permeability graphite, and a siliconized silicon carbide-coated graphite have been degassed. In addition to the volume and composition of the gas evolved by the various graphite samples, data have been obtained for several samples that relate the volume of gas evolved to the time of degassing at 600 and 1000°C.

The graphite samples examined during this period included:

1. SP-AGOT, a National Carbon Company specially purified AGOT grade of graphite having a very low ash content,
2. AGOT-NC6, a 16- by 16-in. extrusion (that is, an extrusion of the size specified for the EGCR graphite columns) prepared by the National Carbon Company from Texas coke having a maximum grain size of 0.03 in. (Hanford designation, NC6),
3. AGOT-VCB, a 4 1/2- by 4 1/2-in. extrusion prepared by National Carbon Company from a regular particle size mix of Great Lakes Continental needle coke that was AGOT processed (Hanford identification, VCB).
4. HPDA, a 4 1/2- by 4 1/2-in. extrusion prepared by Great Lakes Carbon Corporation from a regular particle size mix of Continental needle coke (Hanford identification, GL-11).
5. N964, a 4 1/2- by 4 1/2-in. extrusion prepared by the Speer Carbon Company using Great Lakes Continental needle coke in a regular particle size mix (Hanford designation, SP23B).
6. R-0025, a National Carbon Company low-permeability molded graphite having a density of $\sim 1.88 \text{ g/cm}^3$.

The results of the current degassing studies, in which the various graphite samples were heated to 1000°C by using external resistance heating, are presented in Table 6.8. The temperatures listed in the table are the temperatures of gas collection, and the gas volume listed at a given temperature represent the volume of gas evolved between that temperature and the next lower given temperature. (The 300°C value gives the volume of gas evolved between room temperature and 300°C). The results obtained for Great Lakes R-6HLM graphite (now designated R-2) are in general agreement with the values reported previously¹¹ for this

¹¹"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, p. 198.

Table 6.8. Volume and Composition of Gas Evolved by 1 1/2-in.-diam, 2-in.-long Graphite Specimens During Heating to 300, 600, and 1000°C

Graphite Sample	Temperature (°C)	Time (hr)	Gas Volume (cm ³ /100 cm ³ of graphite)	Gas Constituents											
				H ₂		Hydrocarbons		H ₂ O		CO ₂		N ₂		CO	
				Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³
Great Lakes	300	3	0.7	2		8		48	0.3	6		13	0.1	2	
R-6HLM	600	8	3.7	8	0.3	12	0.4	17	0.6	42	1.5	4	0.1	16	0.6
No. R-2 ^a	1000	12	13.7	22	3.1			3	0.4	9	1.3	2	0.2	64	8.8
	Total		18.1		3.4		0.4		1.3		2.8		0.4		9.4
National Carbon	300	4	0.3			31	0.1	36	0.1	8		20	0.1	4	
SP-AGOT	600	11	1.0	3		15	0.2	16	0.2	16	0.2	5	0.1	44	0.4
No. 1	1000	7	3.3	37	1.2	1		6	0.2	2	0.1	3	0.1	52	1.7
	Total		4.6		1.2		0.3		0.5		0.3		0.3		2.1
SP-AGOT	300	4	0.4	0.8		33	0.1	32	0.1	6		21	0.1	5	
No. 2	600	12	1.2	4		20	0.3	19	0.2	16	0.2	4		36	0.5
	1000	9	4.6	67	3.1	1		3	0.2	0.8		3	0.1	21	1.0
	Total		6.2		3.1		0.4		0.5		0.2		0.2		1.5
SP-AGOT	300	4	0.3	4		25	0.1	45	0.1	4		17	0.1	5	
No. 3	600	10	1.5	6	0.1	21	0.3	22	0.3	14	0.2	3		35	0.6
	1000	7	4.2	77	3.2	1		2	0.1	0.5		3	0.1	17	0.7
	Total		6.0		3.3		0.4		0.5		0.2		0.2		1.3
AGOT-NC6	300	4	0.5			7		73	0.4	5		14	0.1	2	
No. 3	600	17	6.3	5	0.3	9	0.6	30	1.9	46	2.9	2	0.1	7	0.5
	1000	21	12.4	40	4.9	1	0.1	1	0.1	2	0.2	6	0.7	52	6.4
	Total		19.2		5.2		0.7		2.4		3.1		0.9		6.9

^aAnalysis of gas evolved during heating to 300°C showed, in addition to the constituents indicated, 13% O₂, 5% H₂S, and 2% SO₂.

Table 6.8 (continued)

Graphite Sample	Tempera- ture (°C)	Time (hr)	Gas Volume (cm ³ /100 cm ³ of graphite)	Gas Constituents											
				H ₂		Hydrocarbons		H ₂ O		CO ₂		N ₂		CO	
				Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³
AGOT-NC6 No. 8	300	8	0.5			0.3		10		0.7		64	0.4	8	
	600	10	6.5	7	0.5	12	0.7	32	2.1	41	2.6	3	0.2	6	0.4
	1000	24	12.9	56	7.3	1	0.1	0.5	0.1	1	0.2	5	0.7	36	4.6
	Total		19.9		7.8		0.8		2.2		2.8		1.3		5.0
AGOT-VCB No. 1	300	5	0.5	4		12		52	0.3	8		21	0.1	2	
	600	11	10.9	4	0.4	4	0.4	10	1.1	66	7.2			15	1.7
	1000	11	6.4	45	2.9	0.6		1	0.1	9	0.6	0.5		43	2.8
	Total		17.8		3.3		0.4		1.5		7.8		0.1		4.5
AGOT-VCB No. 2	300	13	0.5	10	0.1	10		60	0.3	5		13	0.1	2	
	600	11	11.2	3	0.3	5	0.5	6	0.7	70	7.8	0.4	0.1	15	1.7
	1000	10	7.5	45	3.4	0.6		1		9	0.7	1	0.1	43	3.2
	Total		19.2		3.8		0.5		1.0		8.5		0.3		4.9
AGOT-VCB No. 3	450	10	1.5	5	0.1	25	0.3	44	0.7	0.7		3		12	0.2
	600	11	9.4	4	0.4	4	0.4	4	0.4	73	6.9	3	0.3	13	1.2
	800	10	3.6	37	1.3	3		1		33	1.2	1		32	1.2
	1000	11	4.4	85	3.7	1		2	0.1	0.1		0.2		11	0.5
	Total		18.9		5.5		0.7		1.2		8.1		0.3		3.1
Great Lakes HPDA No. 1	300	5	0.5	3		6		54	0.3	5		23	0.1	3	
	600	10	1.6	19	0.3	23	0.4	26	0.4	9	0.2	8	0.1	13	0.2
	1000	12	6.2	57	3.5	2	0.1	1	0.1	2	0.1	1	0.1	37	2.3
	Total		8.3		3.8		0.5		0.8		0.3		0.3		2.5

Table 6.8 (continued)

Graphite Sample	Temperature (°C)	Time (hr)	Gas Volume (cm ³ /100 cm ³ of graphite)	Gas Constituents											
				H ₂		Hydrocarbons		H ₂ O		CO ₂		N ₂		CO	
				Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³
HPDA No. 3	300	4	0.5	3		16	0.1	36	0.2	7		29	0.2	6	
	600	18	1.9	9	0.2	29	0.5	21	0.4	14	0.3	10	0.2	18	0.3
	1000	11	4.8	59	2.8	1	0.1	1		3	0.1	1	0.1	36	1.7
	Total		7.2		3.0		0.7		0.6		0.4		0.5		2.0
Speer N964 No. 1	300	16	0.3			13		31	0.1	8		41	0.1	7	
	600	12	1.5	16	0.2	34	0.5	18	0.3	9	0.1	5	0.1	18	0.2
	1000	18	4.9	75	3.7	3	0.1	1		0.3		1		20	1.0
	Total		6.7		3.9		0.6		0.4		0.1		0.2		1.2
N964 No. 2	300	5	0.4			18	0.1	34	0.1	8		32	0.2	6	
	600	17	1.7	12	0.2	34	0.6	12	0.2	9	0.2	16	0.3	16	0.3
	1000	21	4.9	71	3.5	1		2	0.1	0.5		2	0.1	24	1.2
	Total		7.0		3.7		0.7		0.4		0.2		0.6		1.5
National Carbon R-0025 No. E-2	300	11	0.4	3		7		36	0.1	11		36	0.2	7	
	600	23	1.7	16	0.3	25	0.4	19	0.3	16	0.3	7	0.1	17	0.3
	1000	24	7.5	55	4.1	3	0.2	1	0.1	5	0.4	1	0.1	34	2.6
	Total		9.6		4.4		0.6		0.5		0.7		0.4		2.9
R-0025 No. E-3	300	19	0.5	3		9		34	0.2	10	0.1	37	0.2	6	
	600	23	2.1	21	0.4	28	0.6	16	0.3	14	0.3	6	0.1	14	0.3
	1000	29	8.7	54	4.7	2	0.2	0.3		4	0.4	4	0.3	34	3.0
	Total		11.3		5.1		0.8		0.5		0.8		0.6		3.3
3M Coated* No. 4 and 5	300	3	(0.2)	Not analyzed, included in 600°C sample.											
	600	21	0.6	33	0.2	16	0.1	19	0.1	16	0.1	6		11	0.1
	1000	22	3.6	81	2.9	7	0.2	1		0.2		0.4		11	0.4
	Total		4.2		3.1		0.3		0.1		0.1				0.5

*Specimens 1 1/4 in. in diameter and 1 1/4 in. long.

grade of graphite. The total volume of $\sim 6 \text{ cm}^3$ of gas per 100 cm^3 of graphite evolved by SP-AGOT graphite during heating to 1000°C is as small as has been observed for any graphite. The small volume evolved correlates with the very low ash content of this specially purified material. The results given for AGOT-NC6 graphite indicate that this large extrusion prepared from Texas coke evolved somewhat less gas when heated to 1000°C than did the $4 \frac{1}{2}$ - by $4 \frac{1}{2}$ -in. extrusions of National Carbon AGOT graphites examined earlier.

The results given in Table 6.8 for the graphites prepared by the various manufacturers from needle coke are interesting. The behavior of HPDA graphite was quite similar to that of N964 graphite, and it is to be noted that these two graphites were prepared by Great Lakes Carbon Corporation and the Speer Carbon Company, respectively, from the same batch of Continental needle coke. Both graphites evolved 6 to 8 cm^3 of gas per 100 cm^3 of graphite, and, in addition, the composition of the evolved gas was similar, as were the volumes evolved at 300 , 600 , and 1000°C . The ratio of the volume of H_2 to $\text{CO} + \text{CO}_2$ was greater than one, and more than half of the total volume of gas evolved came off during heating from 600 to 1000°C .

The behavior of AGOT-VCB graphite, which was prepared by National Carbon Co. from Continental needle coke from a different batch, was quite different from the behavior of HPDA and N964 graphites. The AGOT-VCB graphite evolved a total of $\sim 18 \text{ cm}^3$ of gas per 100 cm^3 of graphite upon heating to 1000°C and the ratio of H_2 to $\text{CO} + \text{CO}_2$ in the gas was much smaller than one. The volume evolved between 300 and 600°C was greater than half of the total evolved and was considerably greater than the volume evolved between 600 and 1000°C . The large volume of gas evolved between 300 and 600°C contained very little H_2 ; it was predominantly CO_2 . The reason or reasons for the difference in the degassing behavior of these graphites are not known. The AGOT-VCB graphite was, as mentioned above, prepared from Continental coke from a different batch, but differences in manufacturing techniques were probably mainly responsible for the differences in degassing behavior. It is apparent, however,

that all graphites prepared from needle coke do not behave similarly when degassed.

The results given in Table 6.8 for R-0025, a low-permeability graphite, show the evolution of $\sim 10 \text{ cm}^3$ of gas per 100 cm^3 of graphite during heating to 1000°C and indicate that the bulk of the gas was evolved during heating from 600 to 1000°C . The ratio of H_2 to $\text{CO} + \text{CO}_2$ in the evolved gas was somewhat greater than one. Two siliconized silicon carbide-coated graphite specimens obtained from Minnesota Mining and Manufacturing Co. (designated 3M coated in Table 6.8) were outgassed together. The total volume of evolved gas ($\sim 4 \text{ cm}^3$ of gas per 100 cm^3 of graphite) was smaller than observed for coated samples outgassed previously.¹¹ The evolved gas was predominantly H_2 which diffused through the coating; only small amounts of CO and CO_2 were collected. Data on the gas evolved during induction heating of one of these coated specimens to 1500°C are given in Table 6.9. As may be seen, relatively little gas was evolved, even though the coating separated and cracked.

The results of studies of the degassing behavior of the various graphites during induction heating to 1800°C are presented in Table 6.9. In most instances, the evolved gas was held over the specimen during heating from room temperature to 1000°C . After heating for approximately 1 hr at 1000°C , the gas was pumped off while the specimen was held at 1000°C . The collection at 1400°C includes the gas pumped off while raising the temperature from 1000 to 1400°C plus that removed during a holding period at 1400°C . Similarly, the 1800°C fraction was collected while heating from 1400 to 1800°C as well as at 1800°C . In a few cases, where data are given for only 1800°C , the specimen was heated to 1600°C with the gas over it, the gas was pumped off, and the temperature was raised to 1800°C while pumping was continued.

The data given in Table 6.9 show that SP-AGOT graphite evolved $\sim 20 \text{ cm}^3$ of gas per 100 cm^3 of graphite and that the evolved gas was mainly H_2 . The volume of gas evolved is approximately one-half the lowest found for any graphite sample previously examined. The results obtained for AGOT-NC6 graphite were not precise, but it appears that the

Table 6.9. Volume and Composition of Gas Evolved by 1 1/4-in.-diam, 1-in.-long Graphite Specimens During Heating to 1000, 1400, and 1800°C

Graphite Sample	Temperature (°C)	Time (hr)	Gas Volume (cm ³ /100 cm ³ of graphite)	Gas Constituents											
				H ₂		Hydrocarbons		H ₂ O		CO ₂		N ₂		CO	
				Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³
National Carbon	1000	7	10.6	78	8.3	3	0.3	0.7	0.1	0.7	0.1	1	0.1	16	1.8
SP-AGOT	1400	11	10.6	76	8.1	0.4		0.9	0.1	0.1		3	0.3	19	2.1
No. 8	1800	3	2.1	59	1.4	0.1				0.2		4	0.1	37	0.9
	Total		23.3		17.8		0.3		0.2		0.1		0.5		4.8
SP-AGOT	1000	5	10.5	69	7.3	4	0.4	1	0.1	0.7	0.1	2	0.2	22	2.4
No. 9	1400	11	9.2	81	7.4	0.1		2	0.2	0.1		3	0.3	14	1.3
	1800	2	1.1	40	0.4					0.4		10	0.1	47	0.5
	Total		20.8		15.1		0.4		0.3		0.1		0.6		4.2
SP-AGOT	1800	7	22.1	84	18.7	0.4	0.1	0.3	0.1	0.2		0.3	0.1	14	3.1
No. 7															
AGOT-NC6	1000	7	47	57	27	2	0.9	0.7	0.3	0.1		2	1.1	38	18
No. 2A	1400	12	24	73	18	2	0.4	0.2		0.2		2	0.5	23	5.6
	1800	17	16	73	12	1	0.2					4	0.6	22	3.6
	Total		87		57		1.5		0.3				2.2		27.2
AGOT-NC6	1800	11	64	64	41					0.1	0.1	3	1.9	34	22
No. 2C															
AGOT-NC6	1800	16	52	62	32	1	0.5	0.2	0.1	0.2	0.1	5	2.6	31	16
No. 1C															
AGOT-VCB	1000	4	35.9	30	10.6			0.4	0.1	1.0	0.4	1	0.3	68	24.4
No. 4	1400	6	6.6	18	1.2	1	0.1	1	0.1	0.9	0.1	18	1.2	60	4.0
	1800	3	3.1	71	2.2	0.5				0.3		17	0.5	10	0.3
	Total		45.6		14.0		0.1		0.2		0.5		2.0		28.7

Table 6.9 (continued)

Graphite Sample	Temperature (°C)	Time (hr)	Gas Volume (cm ³ /100 cm ³ of graphite)	Gas Constituents											
				H ₂		Hydrocarbons		H ₂ O		CO ₂		N ₂		CO	
				Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³	Vol %	cm ³
AGOT-VCB No. 5	1000	5	38.5	30	11.3	0.5	0.2	1	0.4	1.1	0.4	1	0.4	67	25.8
	1400	7	9.3	35	3.2	0.7	0.1	1	0.1	0.7	0.1	17	1.5	46	4.3
	1800	8	10.5	79	8.3	0.7	0.1			1.2	0.1	7	0.6	11	1.1
	Total		58.3		22.8		0.4		0.5		0.6		2.5		31.2
AGOT-VCB No. 6	1000	3	37.8	28	10.4	0.5	0.2	0.2	0.1	0.2	0.1	1	0.4	70	26.6
	1400	6	7.9	32	2.5	0.7	0.1	0.4		0.3		13	1.0	53	4.2
	1800	5	9.1	77	7.0	0.8	0.1			0.1		7	0.7	14	1.2
	Total		54.8		19.9		0.4		0.1		0.1		2.1		32.0
Great Lakes HPDA No. 4	1000	7	16.1	65	10.5	2	0.3	0.9	0.1	0.7	0.1	1	0.2	30	4.8
	1400	16	21.4	43	9.1	0.7	0.1	0.6	0.1	0.2		4	0.8	52	11.1
	1800	4	10.2	70	7.2	0.7	0.1			0.1		7	0.7	21	2.1
	Total		47.7		26.8		0.5		0.2		0.1		1.7		18.0
HPDA No. 5	1800	21	41.2	66	27	0.8	0.3	0.3	0.1	0.3	0.1	4	1.6	29	12
Speer N964 No. 3	1000	7	19.8	76	15.1	2	0.4	0.6	0.1	1.0	0.2	1	0.2	19	3.5
	1400	12	12.5	59	7.4	0.4	0.1	0.6	0.1	0.1		6	0.7	34	4.3
	1800	11	12.7	56	7.1	0.4	0.1					5	0.7	38	4.8
	Total		45.0		29.6		0.6		0.2		0.2		1.6		12.6
N964 No. 4	1800	19	55	71	39	0.4	0.2	0.4	0.2	0.2	0.1	2	1	25	14
3M Coated No. 4	1500	7	3.3	51	1.7	24	0.8			0.9		11	0.4	13	0.4

total volume of gas evolved to 1800°C by this graphite is comparable to that found earlier for National Carbon AGOT-III graphite. The proportion of H₂ in the evolved gas was found to be larger, however, in the case of AGOT-NC6.

The results given in Table 6.9 for the three graphites prepared from needle coke, namely, HPDA, AGOT-VCB, and N964, show that these three graphites evolved approximately equivalent volumes of gas upon heating to 1800°C. The gas evolved by HPDA and N964 contained a larger proportion of H₂, however, than the gas obtained from AGOT-VCB. If it is assumed that the degassing was essentially complete upon heating to 1800°C, the volume found for AGOT-VCB by heating to 1000°C by external resistance heating, as given in Table 6.8, is roughly one-third the total gas content of the graphite; the corresponding fraction for the HPDA and N964 graphites is only about one-sixth. The AGOT-VCB graphite evolved about two-thirds of its total gas content when heated to 1000°C by induction heating, whereas the HPDA and N964 graphites evolved less than one-half of their total gas when heated under similar conditions. A comparison of the volumes of gas evolved through 1000°C by external resistance and induction heating shows that the induction heating results in the evolution of at least twice as much gas as does external resistance heating at a common temperature. The reason for this difference is not known, but this behavior has been observed consistently for all the graphites examined.

Data are plotted as Figs. 6.1, 6.2, and 6.3, which show for several graphites the relationship of the total volume of gas evolved at 600 and 1000°C to the logarithm of the time of heating. Values for the volume evolved during the first 30 or 60 min are not reliable, if appreciable volumes of gas are involved, for two reasons: (1) gas is evolved while the specimen is coming up to temperature, and (2) the rate of evolution may be so high upon attainment of temperature that the Toepler pump is unable to remove the gas as rapidly as it is evolved. Consequently, no values are reported for times less than 30 min unless the initial rate of evolution was low.

Fig. 6.1. Volume of Gas Evolved by TSF, AGOT-NC6, and R-6HLM Graphites Plotted Against Log of Time.

It may be seen that plots of the volume of gas evolved at 1000°C against the log of time result in essentially straight lines for the several graphites examined over periods of time ranging from 1 to 2 hr up to ~20 hr. The results obtained for gas evolution at 600°C are not, however, as consistent when handled in this manner. For example, N964, TSF, and R-0025 graphites yielded data which when plotted as indicated gave essentially linear relationships. AGOT-NC6 and AGOT-VCB graphites gave results at 600°C, on the other hand, which unquestionably depart from linearity. The behavior of AGOT-VCB is of particular interest because, in addition to the curve for 600°C not being linear, it lies well above the 1000°C curve. The curves given for AGOT-VCB-2 and -3 at 600°C, while similar, are not strictly comparable. The No. 2 sample had been degassed at 300°C and the No. 3 sample had been degassed at

Fig. 6.2. Volume of Gas Evolved by AGOT-VCB and N964 Graphites Plotted Against Log of Time.

Fig. 6.3. Volume of Gas Evolved by R-0025 Graphite Plotted Against Log of Time.

450°C prior to degassing at 600°C. This difference in treatment accounts for the displacement of the No. 3 sample curve to lower values. The sample degassed at 1000°C to yield the data for the No. 3 line had been degassed at 800°C. If instead it had been degassed at 600°C, the line would have fallen where the No. 1 line is, and a definite crossing of the 600°C curve would have occurred. The only apparent explanation for the difference in the behavior of the graphite lies in the fact that upon degassing at 600°C the AGOT-VCB and -NC6 samples evolved gas having a high CO₂ concentration, whereas N964, R-0025, and TSF samples gave gas having a much lower CO₂ concentration and a higher H₂ concentration. This in no sense explains why AGOT-VCB released a larger volume of gas between 300 and 600°C than between 600 and 1000°C in contrast to the behavior of AGOT-NC6 and virtually all other graphites examined. Obviously, the surface oxide is destroyed more readily and this, in turn, must be related to the inherent structure of this particular graphite.

Fig. 6.3 gives the results of duplicate determinations on R-0025 graphite. Good precision was obtained in this case, with straight line relationships existing at both 600 and 1000°C.

Transport of Gases Through Graphite

Experimental studies of the interdiffusion of argon and helium¹² through an AGOT reactor-grade graphite septum¹³ under uniform total pressure conditions have been extended to include a series of experiments at 100°C. Previously reported¹²⁻¹⁴ permeability data, pore-size distribution curves, and the initial interdiffusion experiments indicated that Knudsen diffusion effects do not contribute to the over-all diffusion mechanism in AGOT graphite. Conclusions regarding the nature of the mechanism cannot be made at this time because an unexpected drift of

¹²"GCR Quar. Prog. Rep. Dec. 13, 1959," ORNL-2888, p. 159.

¹³"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, p. 201.

¹⁴J. Truitt et al., "Transport of Gases Through Ceramic Materials," ORNL-2931, p. 149.

molecules has been observed at uniform pressures for both types of graphite studied thus far, Speer No. 1 and National Carbon AGOT. The drift of molecules is such that, as shown in Fig. 6.4, all ratios of the apparent coefficients for helium to those for argon at equivalent conditions of temperature and pressure are approximately equal to the square root of the ratio of the molecular weight of argon to that of helium. These are precisely the results that would be expected if a parallel Knudsen diffusion mechanism were controlling; however, this possibility must be ruled out on the basis of the evidence previously cited,¹²⁻¹⁴ which has been strengthened by the data obtained recently at 100°C. The over-all steady-state diffusion behavior may be described by the expression

$$n_1 = D_1 \frac{A}{L} \frac{P}{RT} \Delta N_1, \quad (1)$$

where D_1 is an apparent diffusion coefficient, A/L is the area-to-length ratio of the septum, P/RT is the molar density of the gas, and ΔN_1 is the mole fraction change of component 1 across the septum. This equation is convenient but not precise, since the net drift leads to a nonuniform concentration profile if it must be assumed that all channels are engaged in the same mechanism.

Fig. 6.4. Apparent Diffusion Coefficients for Helium and Argon In AGOT Graphite at Uniform Total Pressure.

Nonlinear concentration profiles may readily be taken into account by defining the net drift, $\bar{n}_T$, as the vector sum of $\bar{n}_1$ and $\bar{n}_2$. The expression given by Present¹⁵ and utilized by Weissberg¹⁶ for the total flow of either component (in this case helium) is then applicable:

$$\bar{n}_1 = N_1 \bar{n}_T - D_{12} A \frac{P}{RT} \frac{\partial N_1}{\partial x} , \quad (2)$$

where D_{12} is the mutual diffusion coefficient. Integration of Eq. (2) in terms of the concentrations at $x = 0$ and $x = L$ gives

$$(D_{12})_{\text{eff}} \ln \frac{[D_1 - (D_1 - D_2)N_1, x=L]}{[D_1 - (D_1 - D_2)N_2, x=0]} = (D_1 - D_2) \Delta N_1 . \quad (3)$$

The apparent diffusion coefficients may then be converted to standard mutual diffusion coefficients, D_{12} , which are the same for both gases. Calculations based on the data of Fig. 6.4 and Eq. (3) resulted in the data of Fig. 6.5.

¹⁵R. D. Present, "Kinetic Theory of Gases," p. 49, McGraw-Hill, New York, 1958.

¹⁶H. L. Weissberg and A. S. Berman, "Diffusion of Radioactive Gases Through Power Reactor Graphites," ORGDP-KL-413 (April 6, 1959).

Fig. 6.5. Mutual Diffusion Coefficients Obtained for Helium-Argon Mixtures in AGOT Graphite in Uniform-Total-Pressure Experiments.

In order to test the equation and the assumed model with respect to variations in $\bar{n}_T$, another series of experiments was performed at 100°C with a slightly higher pressure on the argon side than on the helium side. The results of these experiments are compared in Fig. 6.6 with the corresponding uniform pressure results. The D_1 values decreased and the D_2 values increased, although the D_{12} values for both series of experiments remained the same. Thus it appears that Eq. (3) is indeed adequate for correlating the data in terms of an effective mutual diffusion coefficient for the types of graphite under consideration.

An examination of the manner in which the effective D_{12} values vary with temperature and pressure reveals that the classical formulation for helium-argon mixtures¹⁷ is followed; that is,

$$(D_{12})_{\text{eff}} = (D_{12})_{0,\text{eff}} \frac{P_0}{P} \left(\frac{T}{T_0} \right)^{1.75} \quad (4)$$

¹⁷S. Dushman, "Scientific Foundations of Vacuum Techniques," p. 77, John Wiley, New York, 1949.

Fig. 6.6. Diffusion Coefficients for Helium and Argon in AGOT Graphite at 100°C.

The degree to which Eq. (4) is followed is demonstrated by the proximity of the normalized coefficients to the solid horizontal line of Fig. 6.7.

At this stage of the investigation, it may be concluded that the total transport of either gaseous constituent depends on two flow components: (1) a net drift that can be varied by imposing small pressure drops across the septum but which is not zero under uniform pressure conditions, and (2) a classical diffusion term which differs from hard-book values only through the internal geometry of the graphite.

Fig. 6.7. Mutual Diffusion Coefficients for Helium-Argon Mixtures in AGOT Graphite Normalized to 50°C and 2 atm.

Diffusion of Nonvolatile Fission Products in Graphite

A study has been initiated of the mechanisms involved in the release of fission products from unclad high-temperature fuel elements in a gas-cooled reactor. The release of the fission-products from the UO₂ fuel particles will occur as a multistep mass-transfer process in graphite or other moderator matrix. The two principal mechanisms of fission-product release from UO₂ are solid-state diffusion from the fuel lattice and recoil of the fission fragments into the moderator matrix with a range

of about 10^{-4} cm.¹⁸ The percentage of fission fragments that recoil into the matrix varies with the particle size of the UO_2 and the physical properties of the matrix. For 100- μ UO_2 particles, the recoil can be 7% or more of the total amount released.¹⁸ In graphite the fission products can migrate in the crystallites on the free surfaces of the matrix or inside the pores.

Solid-state diffusion in the crystal lattice along the dislocations or between the crystallites has been studied in one set of experiments. By utilizing an infinite plane geometry, rates of diffusion of individual fission products into AGOT graphite were measured at temperatures between 750 and 1200°C. Agreement of the data obtained with the earlier work of Findlay¹⁹ was good. As indicated in Fig. 6.8, the coefficients of diffusion for the fission products studied decreased with increasing atomic radii. This was anticipated based on the diffusion taking place as a random walk process.²⁰ Then

$$D = \frac{1}{2} \sum \Gamma_i (\Delta x_i)^2 ,$$

where Δx_i is the distance between jumps. The rate of jumping is

$$\Gamma_i = \frac{kT}{h\nu} e^{-\Delta V/kT} ,$$

where

$$\Delta V = (A/r) + (B/r^6)$$

¹⁸T. A. Lane et al., "A Study of Problems Associated with Release of Fission Products from Ceramic Fuels in Gas-Cooled Reactors," ORNL-2851, October 1959.

¹⁹T. R. Findlay, "Diffusion of Fission Products from Graphite at 800°C," AERE-C/R-2683.

²⁰G. Toos, "Theoretical Physics," 2nd ed., Hafner Publishing Co., New York, 1950.

Fig. 6.8. Diffusion of Fission Products in Graphite as a Function of the Atomic Radii.

is the energy required for fixing the atom in the matrix. The relationship between the coefficients of diffusion and the atomic radii follow readily from these expressions.

Another group of experiments was initiated to study over-all migration rates of individual fission-product elements in AGOT graphite at temperatures between 1200 and 1900°C. The experimental approach is to measure the release rates for radial diffusion from a central cylindrical source through an enclosed hollow cylinder heated in a high-temperature vacuum furnace, followed by determination of the concentration profile. Diffusion from SrO , BaO , CeF_3 , Y_2O_3 , ZrO_2 sources through AGOT graphite cylinders is being studied.

Although the experimental data are incomplete, indications of significant changes in the mass-transport mechanisms as a function of temperature have been obtained. Rates of migration are much higher, increasing by about 10^5 from 800 to 1500°C. This indicates significantly decreased energies of activation and movement, at least partially, in the open pores. The increases in rates are not necessarily of the same order of magnitude for the different diffusing species. Yttrium and zirconium react with the graphite to form stable carbides. As a result the release rates are below 8%. On the other hand, barium is released rather rapidly, with less than 5% remaining after heating to 1500°C for 16 hr.

The experimental work has indicated the feasibility of a generalized correlation for solid-state diffusion of fission products at temperatures below 1000°C. The experimental data for the 1300 to 1900°C range indicate that the mechanisms of mass transport are not the same in this range as at lower temperatures. Rapid fixation of elements that form stable carbides at these temperatures takes place and the migration of others that do not form stable nonvolatile compounds with the matrix is accelerated.

Chemical Behavior of Fission Products in Fissioning UO_2

It is evident that a cubic UO_2 phase containing ZrO_2 , SrO , BaO , Y_2O_3 , La_2O_3 , and rare earth oxides in solid solution will exist in EGCR fuel capsules as the materials listed "grow in" during fissioning of the fuel. This phase will not melt, however, at the expected operating temperatures of the EGCR fuel material.

In the previous report,²¹ it was assumed that fission-product molybdenum would compete successfully for oxide ions released by fission and that MoO_2 would be present in the UO_2 fuel. A recent British Patent²²

²¹"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 209-11.

²²British Patent 809,693.

indicates, however, that UO_2 will reduce molybdenum oxides to metallic molybdenum. Since the reduction reaction is presumed to be

the reduction would not be expected to proceed if the oxygen-to-uranium ratio exceeded a certain value. Although the limiting value of this ratio has not been well defined, it appears that fission-product molybdenum will appear as metal in the EGCR fuel.

Molybdenum, cesium, rubidium, technetium, ruthenium, rhodium, and palladium, which will exist as metals, have a combined yield of about 65 atoms per 100 fissions. At 10 000 Mwd/T this corresponds to about 4 g (perhaps 0.5 cm³) of metal per fuel capsule. It is possible that these metals will form a liquid alloy at the temperatures anticipated within the fuel element. It seems likely, but not certain, that this alloy would be largely retained within the porous fuel matrix. If the alloy dripped from the fuel, it would almost certainly damage the stainless steel fuel can through alloy formation.

Purification of Cooling Gases

Methods of removing impurities, particularly radioactive fission-product gases, from the coolants of gas-cooled reactors are being investigated. Additional measurements of dynamic adsorption on charcoal have been made, and a generalized Oracle program for adsorber evaluation was written.

For optimum design of an adsorber for the removal of noble-gas fission products from helium, dynamic adsorption data for the actual adsorbents and for the actual operating conditions are required. A fair quantity of more or less applicable data for Columbia G activated carbon, 8/14 mesh, is available, but this material is no longer being manufactured. Measurements are therefore being made of other promising commercial adsorbents. These measurements include the effects of helium pressure and velocity and adsorbent temperature on fission-gas retention time

and on N, the number of theoretical plates in an adsorber. Preliminary data for Columbia HCC activated carbon, 12/28X, are summarized in Table 6.10. These data are currently applicable to the design of adsorbers for GCR-ORR loop experiment No. 2. The data of Table 6.10 were obtained from Kr⁸⁵ elution curves. The dynamic adsorption coefficient, k, which is approximately proportional to retention time, was based, somewhat arbitrarily, on the volumetric flow rate measured at the carrier gas pressure in the adsorber at 24°C. As indicated by the values of k and N, HCC-12/28X charcoal should be a very useful adsorbent for fission-product gases. Possible disadvantages are its small particle size (and the associated pressure drop) and, as with charcoals in general, its combustibility.

In connection with these measurements, the effects of varying the length of pulse injection time and of varying the quantity of krypton in a pulse on the shape of elution curves were briefly investigated at 25°C using Columbia G activated carbon with helium as the carrier gas. It was observed that variations over rather wide limits caused relatively little effect, as measured by N and k, and thus the values presented here are applicable to any of the fission-gas concentrations normally encountered.

A few elution curves were also obtained with hydrogen as the carrier gas. The measurements indicated that data obtained on charcoal at 25°C

Table 6.10. Dynamic Adsorption of Krypton on Columbia HCC Activated Carbon, 12/28X, from Helium Carrier Gas

Adsorber Temperature (°C)	Helium Pressure (psia)	Superficial Helium Velocity (ft/min)	Adsorption Coefficient, k (cm ³ /g)	Number of Theoretical Plates, N, per Foot
24	14.5	1.6	56.6	72
24	325	1.6	53.3	81
24	325	0.5	48.2	151
-33	14.5	1.6	520	103

with helium carrier gas at high pressures would probably be applicable, from a practical standpoint, to hydrogen carrier gas, so long as the other conditions were unchanged.

A charcoal adsorber designed to remove fission products that might be accidentally released into the off-gas system of the GCR-ORR loop was studied. An Oracle program was written for computing the quantity of radioactivity associated with the 48 isotopes assumed to be released through rupture of the capsule assembly. The magnitude of the hazard was then evaluated by calculating the maximum radiation doses to various body organs that could result by such a release to the atmosphere. Several charcoal trap designs were studied by observing the variation in calculated radiation dose as a function of trap efficiency. It was concluded that an off-gas system consisting of an absolute filter and an iodine vapor adsorber would reduce the hazard of an accidental fission-product release to an acceptable level.

Chemical Analyses of Reactor Materials

Analyses of Mixtures of Helium and Argon

In the studies of the diffusion of gases through graphite (discussed above), diffusion rates are derived from measurement of the concentration of argon in helium and helium in argon in effluent streams from a cell in which pure helium and argon flow past a graphite membrane, or septum. The concentration of the diffused species in these samples ranges from a few hundred ppm to 10%. Since the thermal conductivity of helium is more than eight times that of argon, these binary mixtures are particularly suitable for continuous analysis by thermal-conductivity measurements. Accordingly, a thermal-conductivity apparatus has been fabricated from basic components supplied by the Gow-Mac Instrument Company.

Each sample stream is analyzed by comparing its conductivity with that of a pure reference-gas stream in an eight-element thermal-conductivity bridge that is mounted in a thermostatted oil bath maintained at a temperature of 37°C. The eight tungsten filaments are connected

in series to form four arms of a Wheatstone bridge in which gas streams of sample and reference are directed through opposite arms of the bridge to provide a signal four times that generated by a conventional two-element bridge. Bridge current is obtained from a transistorized power supply. The output signal from the bridges is measured with a 1-mv recorder equipped with switches to provide for the recording of a signal from either bridge or from each bridge alternately. A stepwise attenuator provides attenuation up to one hundredfold.

A schematic flow diagram of the system for measuring helium in argon is presented in Fig. 6.9. A complementary arrangement is provided for the measurement of argon in helium. Three-way brass stopcocks are arranged to permit convenient standardization against one of three synthetic gas mixtures which contain approximately 2, 5, and 9% of the minor component, respectively. Four additional stopcocks are provided to direct the flow

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Fig. 6.9. Schematic Flow Diagram of Apparatus for the Determination of Helium in Argon.

through an alternate path for balancing the bridge. When the flow-directing stopcocks are turned to the alternate position, the reference gas is passed through all elements of the bridge and the sample stream is by.. passed through the original sample vent, since it is necessary to maintain a constant flow through the diffusion cell. The flow-directing stopcocks thus permit the balancing of the bridge with only momentary interruption of flow, and they preclude the introduction of air, which might cause overheating and consequent damage to the tungsten filaments.

Empirical calibration is required because the conductivities of mixtures of helium and argon do not vary linearly with composition. For a bridge current of 120 ma, the concentration of helium in argon is given by the expression

$$C = 0.0260_6 V + 0.0000233 V^2 + \dots ,$$

in which C is the concentration of helium in mole per cent and V is the bridge output in millivolts. This relationship holds within a few tenths of a per cent up to concentrations of 10%. The concentration of argon in helium varies linearly up to concentrations of 10% according to the relationship (bridge current, 300 ma)

$$C = 0.0145_4 V + \dots .$$

Full-scale recorder deflection is thus produced by 145 ppm of argon in helium or by 260 ppm of helium in argon, while the range can be extended to a concentration of 10% by means of the stepwise attenuator. For the concentration range of 2 to 10%, the coefficient of variation of a measurement is only a few tenths of a per cent. The precision and sensitivity of the method thus compare favorably with those obtained in analyses of intermittent samples by conventional laboratory methods, such as mass spectrometry or gas chromatography. The accuracy of the method is determined by the accuracy of preparation of the synthetic standards.

Measurements are usually carried out at flow rates between 100 and 300 cm³/min; however, the flow can be reduced to about 20 cm³/min at some sacrifice of response time. The response time is determined by the period required for the flushing of residual gas from the system. In the measurement of concentrations in the per cent range, equilibrium is attained within 3 to 5 min at flow rates of 200 cm³/min. When the concentration is abruptly reduced, as in bridge-balancing, a period of 12 to 15 min is required. While the system was designed for operation at atmospheric pressure, measurements at pressures up to 15 psig have been found to be satisfactory if the pressure is held constant and the apparatus is calibrated under the conditions of the measurement. The apparatus can be used for the analysis of most binary gas mixtures; however, the sensitivity will obviously be reduced for combinations with less favorable thermal-conductivity relationships.

Preparation of Synthetic Gas Mixtures

The increased demand for mixtures of gases for calibration of instruments and for maintenance of atmospheres of controlled composition has necessitated the development of techniques for the preparation of synthetic mixtures in quantities up to 100 ft³. The simple apparatus used for these preparations consists of a high-pressure manifold fitted with a 0 to 2000 psig reducing valve, fittings for two standard 200-ft³ cylinders, and valved ports for pressure gages and evacuation. The manifold and cylinders can be evacuated to pressures of about 1 μ , and test transfers of helium have been carried out with atmospheric contamination limited to about 1 ppm. Pressures are measured with calibrated gages of a specified accuracy of 0.25% or, in cases of maximum dilution, with a mercury barometer. Dilutions in excess of one hundredfold are carried out in two or more steps. On the basis of the quantities measured, a single dilution can be carried out with a relative error less than 0.5%, and the error in dilution to the 10 ppm range is of the order of 1 to 2%.

In the preparation of a desired mixture, an evacuated cylinder is filled to a calculated pressure with the gas or mixtures to be diluted.

The cylinder is then allowed to stand for several hours until the gas is in thermal equilibrium with the cylinder, as indicated by constant-pressure readings. The temperature of the cylinder is measured with a skin thermometer. A cylinder of the diluent is then connected to the manifold, which is then evacuated. Sufficient diluent to yield the calculated total pressure is then rapidly transferred. After the equilibrium pressure of the mixture has been measured, the cylinder is tilted to an angle of about 45 deg, and its lower end is heated with a heat lamp for 24 hr to insure complete mixing by convection.

Factors for correction for the nonideality of the more common gases have been calculated. These corrections, which usually amount to 5% for a single dilution, are calculated from the van der Waals equation, since a comparison of calculations for typical mixtures by the more elaborate equations of state²³ indicates that the simpler corrections are accurate to a few tenths of a per cent.

The mixtures that have been prepared include six-component mixtures of H₂, N₂, CH₄, CO, and CO₂ in concentrations from 1000 ppm to 1% in helium, oxygen in argon, oxygen in helium, carbon monoxide and carbon dioxide in argon, and various mixtures of helium and argon. The precision of this method of preparation is indicated by the data of Table 6.11, which presents calibrations of a thermal-conductivity apparatus with synthetic mixtures of argon in helium.

Sequential Plan for Sampling and Testing Batches of UO₂ Pellets

A sequential plan for sampling and testing batches of UO₂ pellets for potential use in the EGCR was investigated as a means of reducing the number of analyses required per batch to a minimum while also insuring, with a high degree of certainty, that pellets are rejected when the atomic ratio of oxygen to uranium exceeds 2.02. In this sequential scheme, it

²³J. A. Beattie and O. C. Bridgeman, "A New Equation of State for Fluids. II. Application to Helium, Neon, Argon, Hydrogen, Nitrogen, Oxygen, Air, and Methane," J. Am. Chem. Soc. 50, 3133 (1928).

Table 6.11. Calibration of Thermal-Conductivity Apparatus

Calculated Concentration of Argon (mole %)	Conductivity Bridge Signal (mv)	Calibration Factor, Ratio of Argon Concentration to Signal
2.367	162.9	0.01453
5.458	374.9	0.01456
8.714	600	0.01452

is assumed that the pellet material is homogeneous and composed entirely of oxygen and uranium, that the total oxygen content of a pellet is a reliable measure of the atomic ratio of oxygen to uranium in the UO_2 material, and that the total quantity of uranium can be determined with a high degree of precision. Furthermore, certain assumptions were made regarding the risk the consumer was willing to take of accepting batches of UO_2 pellets in which the atomic ratio of over 5% of the pellet was greater than 2.02; this is designated as the consumer's or β risk. Likewise, the risk of rejecting a batch of pellets unnecessarily was considered (producer's, α risk).

This plan is based on a single-sided alternative hypothesis²⁴ in which α and β risks are specified, the precision of the analytical results is known with a high degree of certainty, and provision is made for limiting the number of analyses required to reach a decision. The proposed sampling and testing plan is described schematically in Fig. 6.10.

The adjusted cumulative total, T_0 , is obtained by subtracting a constant (in this case, 88.04) from each observed uranium value and computing the algebraic sum of these differences. The number of analyses required to reach a decision may vary from 2 to 5. If T_0 is equal to or greater than the critical acceptance limit, T_a , after two analyses, that

²⁴Owen L. Davies (ed.), "The Design and Analysis of Industrial Experiments," 2nd ed., p. 60, Hafner Publishing Co., New York, 1956.

Fig. 6.10. Sequential Testing Plan for Batches of UO_2 Pellets: Referee Method for Uranium.

batch of UO_2 can be accepted without additional analyses. Conversely, if T_0 is less than the critical rejection limit, T_R , the batch of UC_2 is rejected at this point. If, however, T_0 is less than T_a but greater than T_R , no decision can be made. In this case, the total uranium content of a third pellet is determined, and a new value is computed for T_0 . This process is continued until T_0 is greater than T_a or less than T_R , or until five pellets have been analyzed. At this point, a decision can be made even if T_0 is less than T_a and greater than T_R ; T_0 is then divided by 5 to give the average adjusted cumulative total, $\bar{T}_0$, and, if $\bar{T}_0$ is greater than T_R for $N = 2$, the batch of UO_2 is accepted; otherwise the batch is rejected. It should be noted that the reliability of this plan for evaluating the acceptability of batches of UO_2 may be seriously

affected if the basic assumptions are not valid, if some critical factor was not considered in the design of this plan, or if a less precise method of analysis is substituted for the one used herein.

Measurement of High Temperatures

Attachment of Thermocouples for Measurement of Surface Temperatures in High-Velocity Cooling-Gas Streams

The investigation of factors influencing the accuracy of thermocouples attached to metal surfaces in high-velocity cooling-gas streams was continued. The previously discussed²⁵ theoretical calculations on the Oracle of heat transfer in a thermocouple junction regarded as a cooling fin were extended to consider the effects of varying other cooling conditions, including the length of exposed lead wire and the thickness of the cooling annulus. Experimental studies of the effects of these variables were also continued.

Development of a High-Temperature Furnace for Thermocouple Studies

High-temperature furnaces are required for use in investigation of thermocouples for use in the temperature range 1000 to 3000°C. For this purpose, furnaces having a relatively small high-temperature zone of the order of 3/8 in. in diameter and 1 in. long are required, with provisions for inserting the thermocouples to be tested and for viewing them with an optical pyrometer under black-body conditions. Several furnace designs have been developed, and all the designs have tantalum or tungsten heating elements supported by copper blocks, usually water-cooled, and are in an inert atmosphere contained in a quartz bell jar. Specially designed high-current, low-voltage transformers are used as the power supply.

One furnace has a heating element made of 0.010-in.-thick tantalum sheet bent into the form of a cylinder 3/8 in. in diameter and 2 1/2 in. long. A slit approximately 1/8 in. wide is left open for pyrometer

²⁵"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 211-17.

viewing. The tantalum tube is resistance-welded to stainless steel rings previously silver-soldered into copper blocks containing water cooling channels. Brass and copper tubing serve the threefold purpose of supporting the water-cooled blocks, supplying coolant to the blocks, and conducting electric current to the heating element. Thermocouples to be tested are inserted through holes in the copper blocks and are suspended near the axis of the tantalum heater. Tantalum foil crimped to a V-shape is attached to the thermocouple junction for black-body viewing. A furnace of this design has the advantage of uniformity of temperature and uniformity of radiation environment surrounding the thermocouple. The interior surface of the tantalum heating element provides a black-body surface for temperature measurement. The maximum operating temperature of a furnace of this design is limited by the mechanical strength of the tantalum heating element. Such furnaces have been operated for brief periods at temperatures up to 2600°C and for periods of several days at 2000°C. In longer periods of operation, serious deformation of the tantalum furnace occurs that leads to nonuniformity of temperature and ultimate failure of the furnace.

Small furnaces consisting of simple tungsten ribbons have been operated up to the melting point of tungsten. Application of furnaces of this type has been limited because of the small high-temperature zone and because of the difficulty in obtaining black-body conditions for measurements of temperatures in the test region.

The most successful high-temperature furnaces have used self-supporting helical heating elements made of tungsten rod 0.040 to 0.125 in. in diameter. Techniques were developed for winding tungsten helices 3/8 to 1/2 in. in diameter and 1/2 in. long. Heating elements of this type are mechanically attached to water-cooled copper blocks of the same type as used in the tantalum furnace described above. Furnaces of this type have operated for periods of several days at over 2800°C without failure.

Quartz bell-jar-shaped enclosures approximately 4 in. in diameter have been used to contain the inert atmosphere (helium) required by

these furnaces. The quartz tube is sealed to a brass base with an O-ring. The system is evacuated to a pressure of 10μ Hg and purged several times with helium before final filling. When atmospheric impurities are present, deposits of yellow or blue tungsten oxide are found on the interior surfaces of the envelope. Under good atmosphere conditions, only a slight black deposit appears on the upper portion of the envelope. The black deposit is metallic tungsten, as evidenced by x-ray diffraction analysis.

The power for these furnaces has been supplied by specially built transformers designed to match the load resistance of the furnaces. The core and winding of a Variac or Powerstat autotransformer are used with the housing and brush assembly removed. The original winding of the autotransformer serves as the primary winding in the high-current transformer. The secondary winding consists of 10 or 20 turns of water-cooled copper tubing wrapped around the toroid core. The ends of this copper tubing are attached to the terminals of the furnace, and the transformer and furnace are a part of the same cooling-water circuit. The transformers of this type have been built using two 50-amp, 115-v Variacs in series. Secondary currents of up to 1000 amp have been obtained at total secondary powers of over 3 kva. These furnaces are now used routinely in the study of high-temperature thermocouples, such as rhenium-tungsten thermocouples with BeO insulation.

Stability of Rhenium Thermocouples

Tests were continued on rhenium-tungsten thermocouples at temperatures from 1000 to 2000°C. Studies are being made of the effects of aging at high temperature in an improved tungsten furnace.

Drift Studies on Chromel-P-Alumel Thermocouples in Various Atmospheres

Tests have been made of Chromel-P-Alumel thermocouples in stagnant helium at 1000°C. In these tests the thermocouples have included a platinum leg as a standard, with the platinum enclosed in unbroken ceramic insulator tubes. The Chromel-P and Alumel wires (dull finish) have been exposed to helium in protection tubes by using 1-in. lengths of ceramic

spaced 0.25 in. apart along the wires. The stagnant helium atmosphere is obtained by applying helium at a pressure of 2 psi to the protection tube. In most cases the helium pressure has been applied to the tube without purging of the original air content to the tube. Quartz protection tubes have been used for all tests. The quartz tubes and ceramic insulators have been baked for several hours at 1000°C in air before use. For some tests, the thermocouple wires have been thoroughly washed with benzene before use. However, there seems to be no difference in results for washed and unwashed wires.

It has been possible to cause rapid negative emf drifts in stagnant helium without foreign metallic contaminants in the protection tube. The inclusion of Inconel, chromium, or type 304 stainless steel filings in a protection tube resulted in negative drifts which were comparable to those of the control thermocouples tested without contaminants. It was noticed that thermocouples tested in the presence of activated charcoal showed higher rates of drift than those tested with metallic contaminants.

Most of the recent work has been with Chromel-P and Alumel without foreign contaminants. It seems that the contaminants may affect the rate of drift, but they are not fundamentally responsible for the drift.

One series of thermocouples was tested to show that the air contained in the protection tube was not important to the negative drift (Fig. 6.11).

Fig. 6.11. Drift of Chromel-P-Alumel Thermocouples in Stagnant Helium.

Several complete thermocouple assemblies that were thoroughly evacuated and purged with helium at room temperature several times during a period of several days were used. These couples showed the same rate of negative drift in stagnant helium as controls with normal air content. During a 24-hr period, all the couples showed a drift of -2.25 ± 0.25 mv. In addition, it was found that exposure of negatively drifting thermocouples to air had no effect on the rate of drift when they were replaced in the furnace under stagnant helium.

The results noted after evacuation of these thermocouples, Fig. 6.11, should be important in explaining the drift mechanism. Thermocouples which were evacuated with a mechanical vacuum pump before insertion in the furnace and were pumped out during the test showed no drift while the pumping was continued. After 18 hr of pumping, helium pressure was applied to the couples and no drift occurred during the next 130 hr.

When negatively drifting thermocouples were evacuated with a mechanical pump, the couples showed a rather sharp rise in emf of about 0.3 mv during a 10-hr period and then remained rather constant in output at the negative error reached. The constancy of output has been noted over a period of about 100 hr in several cases. It has been noted recently

Fig. 6.12. Changes in Resistance of Chromel-P and Alumel Wires under Various Conditions of Thermocouple Application.

Fig. 6.13. Chromel-P vs Alumel Thermal Gradient Profile.

that application of stagnant helium to these couples results in further negative drifts at about the same rate as before evacuation.

Resistance changes of the individual wires during the tests have been measured accurately. As shown in Fig. 6.12, no appreciable resistance changes occurred during negative drifts. This has always been noted, in contrast to increases of resistance of the wires under oxidizing conditions.

Studies of the nonhomogeneity of thermocouple wires after testing for various times in stagnant helium have been conducted. These studies have been carried out by noting the emf output at different positions while slowly removing the thermocouple assemblies from the furnace. After making corrections for the thermal gradient of the furnace, error voltages were plotted against the position of the wire, as shown in Fig. 6.13. In thermocouples showing negative drift, a sharp negative error was found approximately 2 in. inside the furnace block. This position is the point at which the thermal gradient of the furnace becomes very steep. Negative errors as high as 20 mv have been measured, and in all cases the negative errors have been due to nonhomogeneities in the Chromel wires. Errors found in the Alumel wires have always been rather gradual positive errors, with the maximum nonhomogeneity near the hot junction. These errors have usually been found in thermocouples showing positive drift under oxidizing conditions.

It is of interest to note that the Chromel wire becomes shiny and is attracted to a magnet in the region of error. The shiny surface appears to be a film of metal over a layer of black oxide. X-ray emission spectroscopy has shown the film to be over 99% nickel (Chromel-P is 90% Ni and 10% Cr), and metallographic sections have shown the presence of intergranular chromium oxide in this region.

7. DEVELOPMENT OF TEST LOOPS AND COMPONENTS

ORR Gas-Cooled In-Pile Loops

GCR-ORR Loop No. 1

Installation of loop No. 1 and its auxiliary equipment at the ORR is nearing completion. Instrumentation checkout and leak testing of the piping systems are under way. The in-pile section and transfer station have been completed and are ready for installation during the ORR shutdown in July. The equipment needed for the emergency power supply has been obtained.

The thermocouple calibration test has been completed, and the results indicate that fuel element cladding temperatures accurate to $\pm 20^{\circ}\text{F}$ can be obtained. Checkout tests of the fuel element assembly and transfer station equipment and of the insertion and removal procedure were completed. A prototype fuel element assembly was fabricated to test the planned assembly sequence, and the zero fission power element for the shakedown run of the loop in August is being fabricated. Equipment for testing a method of sealing the loop piping with foam plastic has been assembled, and the test is under way. This sealant will be utilized in the event the loop becomes contaminated and must be removed from the ORR pool to prevent contamination of the pool water.

Fabrication of a charged-wire radiation detector similar to that in use by the GE-ANP group at the LITR is under way. This unit will be installed after the initial test runs to provide for the contingency that the originally installed Geiger-Mueller tubes and ionization chambers cannot detect a fission gas leak from the fuel element because of increased loop background radiation.

GCR-ORR Loop No. 2

Design of the primary components of loop No. 2 has been completed, and the evaporator, condenser, full-flow filter, and heater are being fabricated. The design of the side-stream gas cleanup system components

has been completed. The side-stream carbon trap is designed to operate at -30°F , and is to be cooled with liquid Freon-11 circulating through a jacket. The Freon-11 will in turn be cooled by a cascaded mechanical refrigeration system. The weight of the trap plus its shield is within the capacity of the 5-ton crane available at the ORR.

Design has been completed and fabrication is under way on the south-facility plug for placement of the fuel elements adjacent to the ORR core. It is planned to remove the present large south-facility plug and to install the new one during the July shutdown of the ORR.

Design criteria were established for the system required to achieve and maintain a pressure of -1.5 psig in the loop containment cell. This negative pressure will allow a loop rupture to occur and assure that the containment cell pressure will remain below atmospheric for at least 4 days, even if the evacuation pump should stop. The system will consist of an absolute filter, an activated-carbon trap, and a pump for exhausting gas to the ORR off-gas system. The carbon trap is designed to reduce the Kr^{88} activity to 1% of its initial value at an air flow rate of $100 \text{ ft}^3/\text{day}$. A cell leaktightness specification of 1% in-leakage (40 scf) in 24 hr has been established. There will be a permanently installed air-lock system for access to the cell.

The two gas-bearing compressors that will be used in series in the loop have been ordered from Bristol Siddeley Engines Limited of Great Britain.

Remote handling will be routinely employed for removing the test plugs from the containment cells into the shielded carrier. To assist such operation, a remotely operated crane and single-arm manipulator will be mounted on a common set of rails within the cell. These two units have been ordered, and, upon delivery, it is planned to set up the crane and manipulator in a test facility to study the remote operations required for removing the test plug.

A preliminary hazards report on this irradiation facility has been prepared. Tests were run on the analog simulator of the loop to investigate loop accidents and normal transient conditions. The tests showed

that the proposed method of bypassing the cooler for loop control was not adequate. Consequently, control will be based on the heater input power. The seriousness of loss of all helium coolant to the test section was calculated, and it was found that the "after-heat" can be successfully dissipated through the insulation to the moderator coolant without excessively high temperatures in the element. The loop pressure losses were determined for the revised loop piping. There is still about 2.3 psi available for pressure loss across the test element.

EGCR Experiment Loops

Experiment Through-Tube Heat Transfer Analysis

In order to obtain the heat-loss data needed for design of the bellows for the 5 1/2-in.-o.d. (nominal 5-in.-i.d.) through-tubes, the temperature profile along the length of a through-tube was determined. For the analysis, the through-tube was divided into five regions: the bottom nozzle, bottom plenum, core, top plenum, and the top nozzle. The case studied was for no internal heat generation. The heat transfer of the gas film in the top and bottom nozzle sections was assumed to be described by the Dittus-Boelter equation. The gas film heat transfer coefficients on the outside of the tube in the plenum volume were based on gas velocities determined in an EGCR model study¹ at Battelle Memorial Institute. Results of the analysis are presented in Figs. 7.1, 7.2, and 7.3. The reference areas indicated on Figs. 7.1 and 7.2 were used in designing the bellows. The thermal conductivity values for the reflective insulation on the tubes were obtained by extrapolation of experimental data previously obtained.

The results of this study indicate that the tube-wall temperature in the bottom nozzle section will vary from 670 to 770°F under the conditions given on Fig. 7.3 and that the heat losses in the plenum sections

¹L. J. Flanigan, G. R. Whitacre, and H. R. Hazard, "Model Studies of Flow and Mixing in the Partially Enriched Gas-Cooled Power Reactor," BMI-1397.

are a function of the external gas film heat transfer coefficient, as indicated in Figs. 7.1 and 7.2. A summary of the calculated heat losses, by section, is given in Table 7.1.

Table 7.1. Expected Heat Loss from Experiment Loop
Through-Tube Based on an Inlet
Gas Temperature of 950°F

Section of Through Tube	Heat Loss (kw/ft)	Total Heat Loss (kw)
Bottom nozzle	1.2	8.4
Bottom plenum	1.4 (min)	16.8 (min)
	1.64 (max)	19.7 (max)
Top Plenum		
Loop gas at 1100°F and reactor gas at 1050°F	0.22	2.64
Loop gas at 1050°F and reactor gas at 1050°F	0	0
Loop gas at 950°F and reactor gas at 1050°F	0.37 (gain)	4.44 (gain)
Top nozzle	1.51	19.6

The calculated differential thermal expansion of the through-tube, by sections, based on the temperature profile is indicated below:

Section expansion, in.	
Bottom nozzle	0.58
Bottom plenum	0.90
Core	2.52
Top plenum	1.51
Top nozzle	<u>1.14</u>
Gross total expansion, in.	6.65
Less reactor vessel growth, in.	<u>1.98</u>
Net total expansion, in.	4.67

Fig. 7.1. Temperature Profile and Heat Loss of Bottom Plenum Section of a 5 1/2-in.-o.d. Experiment Through-Tube Operating with Helium as the Coolant Gas.

The bellows required for these conditions is described by the following data:

Total axial travel, extension	0
Total axial travel, compression	4.75 in.
Total lateral travel	0.063 in.
Total pressure (either independently)	
External	300 psi
Internal	1000 psi
Life requirement	10 000 cycles

Compressor Head Requirement

Compressor head calculations were made based on loop piping layouts and conceptual component designs that closely approximate the final loop design. The total compressor head required for the loop is determined

Fig. 7.2. Temperature Profile and Heat Loss of Top Plenum Section of a 5 1/2-in.-o.d. Experiment Through-Tube Operating with Helium as the Coolant Gas.

by the head losses occurring in the following loop components: (1) in-pile test section (head loss proportioned so that adiabatic pumping power allocated to this portion of the loop would not exceed 2% of the total loop power in the case of helium or CO₂ cooling and 1% in the case of hydrogen cooling), (2) piping and fittings at 600°F, (3) piping and fittings at 1050°F, (4) six blocking and flow-restricting valves, (5) one diverter valve, (6) one loop cooler, (7) one hot-gas filter, and (8) one electric gas heater.

Total compressor heads were calculated for operation with hydrogen, helium, and CO₂ cooling gas pressures of 500 and 1000 psia. The in-pile test section was assumed to be generating 1500 kw of fission power with a gas inlet temperature of 600°F and an outlet temperature of 1050°F. The case of reduced power operation with a gas inlet temperature of 950°F was also calculated for an assumed constant mass flow rate. In all cases it was assumed that the exit gas from the in-pile test section would be attemperated to 1050°F. The head losses in equipment and piping were

Fig 7.3. Temperature Profile and Heat Loss of Bottom Nozzle Section of a 5 1/2-in.-o.d. Experiment Through-Tube Operating with Helium as the Coolant Gas.

based on incompressible fluid flow. The temperature of the loop coolant from the outlet of the in-pile test section to the inlet of the cooler was taken to be 1050°F; the coolant temperature from the outlet of the cooler to the inlet of the in-pile test section was taken to be 600°F or 950°F; and the gas inside the cooler was taken to be at an arithmetic mean temperature of 825°F or 1000°F. It was further assumed that there was no bypass flow across the compressors.

The calculated system head losses and pressure drops are given in Tables 7.2 and 7.3, and the resulting compressor requirements are shown in Table 7.4. The pressure drop through the test section was conservatively based on the inlet gas density.

The total system pressure drop for operating temperatures other than those of Tables 7.2 and 7.3 can be determined by multiplying the original unit pressure drops by the ratio of the original density to

Table 7.2. Experimental Loop System Head Losses and Pressure Drops for Operation with a Coolant Inlet Temperature of 600°F

Component	For Operation at a System Pressure of 500 psia with Indicated Coolant						For Operation at a System Pressure of 1000 psia with Indicated Coolant					
	H ₂		He		CO ₂		H ₂		He		CO ₂	
	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)
In-pile section	12 200	7.4	8 700	10.6	1 920	26.2	12 200	14.5	8 700	21.2	1 960	53.6
Piping and equipment												
Piping at 600°F	1 180	0.72	2 350	2.86	370	5.08	300	0.36	570	1.40	95	2.54
Piping at 1050°F	430	0.19	870	0.74	150	1.32	110	0.10	210	0.40	40	0.71
Blocking valves at 600°F	520	0.32	1 020	1.24	170	2.33	140	0.16	260	0.63	45	1.17
Diverter valve at 1050°F	1 230	0.54	2 500	2.13	410	3.93	310	0.27	610	1.06	105	1.97
Cooler at 825°F	360	0.18	710	0.72	110	1.35	110	0.11	210	0.42	35	0.80
Filter at 600°F	100	0.07	200	0.24	30	0.34	30	0.03	50	0.13	10	0.15
Heater at 600°F	380	0.23	750	0.92	120	1.65	100	0.12	190	0.46	30	0.86
Total piping and equipment	4 200	2.3	8 400	8.9	1 360	16.0	1 100	1.2	2 100	4.5	360	8.2
Total system	16 400	9.7	17 100	19.5	3 280	42.2	13 300	15.7	10 800	25.7	2 320	61.8

Table 7.3. Experimental Loop System Head Losses and Pressure Drops for Operation with a Coolant Inlet Temperature of 950°F

Component	For Operation at a System Pressure of 500 psia with Indicated Coolant						For Operation at a System Pressure of 1000 psia with Indicated Coolant					
	H ₂		He		CO ₂		H ₂		He		CO ₂	
	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)	Head Loss (ft)	Pressure Drop (psi)
In-pile section	2 700	1.3	1 930	1.7	440	4.4	2 700	2.5	1 930	3.5	450	8.9
Piping and equipment												
Piping at 950°F	1 990	0.93	4 030	3.75	690	6.98	520	0.46	1 040	1.85	170	3.46
Piping at 1050°F	430	0.19	870	0.74	150	1.32	110	0.10	210	0.40	40	0.71
Blocking valves at 950°F	910	0.42	1 810	1.65	320	3.10	230	0.21	450	0.83	80	1.54
Diverter valve at 1050°F	1 230	0.54	2 500	2.13	410	3.93	310	0.27	610	1.06	105	1.97
Cooler at 1000°F	480	0.22	960	0.83	160	1.40	120	0.11	240	0.43	40	0.79
Filter at 950°F	190	0.09	370	0.34	50	0.41	50	0.05	90	0.17	15	0.21
Heater at 950°F	670	0.31	1 330	1.22	230	2.26	160	0.15	330	0.61	50	1.12
Total piping and equipment	5 900	2.7	11 870	10.7	2 010	19.4	1 500	1.4	2 970	5.4	500	9.8
Total system	8 600	4.0	13 800	12.4	2 450	23.8	4 200	3.9	4 900	8.9	950	18.7

Table 7.4. Compressor Requirements for EGCR Experiment Loops
Operating with Hydrogen, Helium, or CO₂ as the Coolant

<u>With Hydrogen as the Coolant</u>				
System pressure, psi	1 000	1 000	500	500
Gas temperature at compressor, °F	600	950	600	950
Mass flow, lb/hr	3 270	3 270	3 270	3 270
System pressure drop, psi	15.7	3.9	9.7	4.0
Compressor head required, ft (based on 1% criterion for pumping power)	13 300	4 200	16 400	8 600
<u>With Helium as the Coolant</u>				
System pressure, psi	1 000	1 000	500	500
Gas temperature at compressor, °F	600	950	600	950
Mass flow, lb/hr	9 190	9 190	9 190	9 190
System pressure drop, psi	25.7	8.9	19.5	12.4
Compressor head required, ft (based on 2% criterion for pumping power)	10 800	4 900	17 100	13 800
<u>With CO₂ as the Coolant</u>				
System pressure, psi	1 000	1 000	500	500
Gas temperature at compressor, °F	600	950	600	950
Mass flow, lb/hr	41 300	41 300	41 300	41 300
System pressure drop, psi	61.8	18.7	42.2	23.8
Compressor head required, ft (based on 2% criterion for pumping power)	2 320	950	3 280	2 450

the new density (evaluated at the new temperature) and summing the pressure drops around the loop. The new head losses can be found by multiplying by the square of the density ratio before summing. If the temperature across the in-pile section is changed, the new head loss and pressure drop can be found by applying the basic equation.

Hydrogen-Cooled Loop Design Study

Requirements were developed for a hydrogen-cooled loop in the EGCR. This loop is to be designed according to the basic criteria used for the design of the helium- and CO₂-cooled loops and is to be ready for operation in the EGCR at the same time. It will be capable of dissipating 1500 kw of power generated in a test fuel element with inlet and outlet temperatures of 600 and 1050°F, respectively, and will be capable of operating at a maximum inlet temperature of 950°F at a reduced power output. The outlet gas will be attemperated if the outlet temperature from the fuel element under test must exceed 1050°F. The loop will be designed to operate at pressures from 500 to 1000 psia.

Compressors for the hydrogen loop were first sized according to the basic design criterion that the pumping power required for the in-pile section should not exceed 2% of the total power output of the in-pile test section. The total compressor head requirement, based on this criterion and the system head losses, is given in Table 7.5. The high head requirement shown by this study proved to be beyond the practical

Table 7.5. Compressor Head Requirements for Hydrogen-Cooled Loop Based on Requirement that Pumping Power Not Exceed 2% of Power Generated

System pressure, psi	1 000	1 000	500	500
Gas temperature at compressor, °F	600	950	600	950
Mass flow, lb/hr	3 270	3 270	3 270	3 270
In-pile section				
Pressure drop, psi	29.0	5.0	14.8	2.6
Head required, ft	24 400	5 400	24 400	5 400
Piping and equipment				
Pressure drop, psi	1.2	1.4	2.3	2.7
Head required, ft	1 100	1 500	4 200	5 900
Total system pressure drop, psi	30.2	6.4	17.1	5.3
Total compressor head required, ft	25 500	6 900	28 600	11 300

range of three of the present He-CO₂ compressors connected in series. The principal limitations of these compressors are impeller speed and size. The feasibility of operation of more than three compressors in series to obtain the required head has not been demonstrated, and extensive testing may be required.

A study of hydrogen cooling was conducted to evaluate the feasibility of the 2% limitation on the pumping power. A comparison was made of head loss and pumping power for the case of an experimental fuel element operating at a constant heat flux per unit area of heat transfer surface when cooled by hydrogen, helium, or CO₂. Since the heat rate for the test element was fixed at 100 kw/ft because of flux peaking considerations, the hypothesis of constant heat flux was reduced to that of constant heat transfer surface per unit length.

The basic equation for pumping power through the test fuel element was developed from the required mass flow and the head loss. The head loss was assumed to consist of a friction loss term and a contraction-expansion loss term. The final requirement on the system was that the heat transport equal the heat transfer. Ratios of the results were taken for comparing the gases. As a further simplification, the ratios were taken for the case of constant heat transfer per unit length, constant average fuel surface temperature, constant temperature difference across the test section, and a constant contraction-expansion loss that was large compared with the friction losses. The results for helium were the reference for the comparisons. In comparing the gases, it was assumed that space was available to adjust the free flow area to the required value. The following equations were used for comparing the free flow area, head loss, and pumping power required for gas x with that for helium (denoted by subscript h):

$$\frac{A_x}{A_h} = \left(\frac{Pr_h}{Pr_x} \right)^{0.4} \cdot \left(\frac{k_x}{k_h} \right)^{0.2} ,$$

$$\frac{h_x}{h_h} = \left(\frac{A_h}{A_x} \right)^2 \frac{(\rho C_p)_h^2}{(\rho C_p)_x^2},$$

$$\frac{W_x}{W_h} = \frac{h_x}{h_h} \frac{(C_p)_h}{(C_p)_x},$$

where

A = free flow area,

Pr = Prandtl number,

k = thermal conductivity,

h = head loss,

ρ = density,

C_p = specific heat,

W = pumping power.

The numerical results of the comparison ratios are presented in Table 7.6. All gas characteristics were evaluated at the average gas temperature. The data show that the pumping power required for the adjusted test fuel element in a hydrogen-cooled loop is 14% of that for a helium-cooled loop and 11 to 14% of that for a CO₂-cooled loop.

Table 7.6. Pumping Power and Head Loss for Hydrogen- and CO₂-Cooled Loops Relative to a Helium-Cooled Loop

Condition		Hydrogen-Cooled Loop Ratios				CO ₂ -Cooled Loop Ratios			
System Pressure (psia)	Average Gas Temperature (°F)	Free Flow Area	Mass Flow	Head Loss	Pumping Power	Free Flow Area	Mass Flow	Head Loss	Pumping Power
500	825	1.13	0.36	0.39	0.14	0.77	4.51	0.29	1.29
500	1000	1.15	0.36	0.38	0.14	0.81	4.37	0.24	1.06
1000	825	1.13	0.36	0.39	0.14	0.76	4.43	0.28	1.23
1000	1000	1.15	0.36	0.38	0.14	0.80	4.32	0.23	1.00

Although this theoretical comparison would not apply to all fuel element test designs, it was considered to be a good indication of the trend in pumping power requirements. It is apparent from this study that the selection of a 2% criterion for helium and carbon dioxide was a reasonable choice. A practical choice for hydrogen, based on the capability of the present helium-carbon dioxide compressors, is 1%. This criterion for hydrogen results in a compressor head requirement which can be satisfied by three compressors in series. It reduces the in-pile section head given in Table 7.5 by approximately 50%. Selecting this criterion provides for considerable variation in head requirements for future test elements.

Kinetics of Experiment Loops

A detailed analog model of the EGCR loops is being developed. Determinations are to be made of the control stability and response for several control schemes, the extent of the pressure and temperature perturbations that would result from several accidents (such as loss of all power, loss of a blower, or loss of cooling water), and the relative importance of the characteristics and heat transfer coefficients of the various components. It appears that the responses of the loops will, in general, be quite sluggish because of the long lengths of heavy-walled pipe between components and the relatively long gas-transit time, of the order of 5 to 8 sec, through the loop.

Component Designs

Gas Filters. The gas filter described previously² is being redesigned based on information obtained from the vendor. It is now proposed to use a round filter, 12 in. in over-all diameter and 11 1/2 in. deep, contained in a 14-in. sched.-80 pipe. This filter can handle helium at a pressure drop of approximately 3.75 in. H₂O and the CO₂ at 1.50 in. H₂O. The resulting pressure drop increase of 1.0 in. H₂O

²"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, pp. 239-42.

compared with that of the filter described previously is negligible in view of the advantages of the smaller sized container.

Gas Heater. As an alternate to the contact type of gas heater described previously,² the feasibility of using a clad element heater is being studied. This type of heater would utilize 45 stainless-steel-clad 1/2-in.-o.d. heater elements. Each element would have a hairpin bend to accommodate thermal expansion. The heating elements would be contained in a pressure vessel 14 in. in outside diameter and approximately 98 in. in over-all length. Several specimens of material and a proposed electric seal assembly have been obtained from a vendor for tests of compatibility with various decontamination solutions.

Loop Valves. After a careful evaluation of the valve proposals submitted by various valve manufacturers, the following three types of valves were obtained for use in screening tests for selecting suitable valves for the loops: a Y-type globe valve with a Limitorque operator manufactured by the Wm. Powell Company, a ball valve with a Limitorque operator manufactured by General Kinetics Corporation, and a gate valve with a hermetically sealed actuator manufactured by Darling Valve and Manufacturing Company.

Leak-Detection System. The EGCR loops will consist essentially of welding piping, but there will be certain items, such as the main-line hot-gas filters, which must be periodically replaced or maintained. It is presently planned therefore to connect such items to the piping system with mechanical joints. One joint being considered is the Conoseal double-gasketed joint developed by the Marman Division of the Aeroquip Corporation. This joint, in common with other types of joints, has some probability of leaking because of a defective gasket, scratches on the seating surfaces, relaxation of gasket seating forces following thermal cycling, misalignment of flanges due to excessive pipe stresses, or other factors. In order to mitigate the consequences of leakage, a study is being made of the problems involved in providing a leak-detection system at each flanged joint for continuous monitoring during loop operation. Systems ranging from those with no provision for leak detection during operation, to those that use a purge gas flowing through the

monitored section, and to those that operate at less than atmospheric pressure or at greater than system pressure have been studied. After considering the various systems, it was decided to use a pressure system for the following reasons.

1. If the purge space were evacuated or supplied with gas at less than atmospheric pressure, the full-system pressure would be imposed on the inner gasket. Leakage from the system process gas would then be outward and, if the gas were radioactive, disposal of the gas would become a problem. Further, a small leak from the inner gasket might become a gross leak due to wire-drawing.

2. With overpressure by a compatible gas, leakage to the process gas would be inward and would be considerably less because of the smaller pressure differential. Operation of the loop could continue under this condition until an orderly shutdown could be affected. Leakage from the outer gasket would be nonradioactive and would, therefore, be nonhazardous.

3. If no leak detection system was provided and reliance was placed on preoperational leak testing of the system as a whole, it appears that a small leak might not be detected when the system was cold and at low pressure and that such a small leak might be serious when the system was hot and at high pressure.

4. The time available for maintenance may be limited during a shutdown, and consequently it is highly desirable to know the location of a leak prior to shutdown.

The precise system, including leak-detection piping, pressure, instrumentation, procedures, etc., necessary to pinpoint the location and magnitude of the leak has not been determined, but studies leading to specification of the system are under way.

Facility Design

The loop supporting facilities are being designed by Kaiser Engineers. Drawings and specifications for portions of the heating and ventilation, loop cooling water, demineralized water, instrument air, and plant air systems have been received. The utilities to be supplied by Kaiser Engineers are indicated below:

Demineralized water

To experimenter's cell	25 gpm
To loop cooler	421 gpm
Total for eight cells	3400 gpm

Service water

To experimenter's cell	20.5 gpm
Total for eight cells	164 gpm

Helium

Volume	To be determined
Supply pressure	2000 psi

Air at a pressure of 120 psi

Instrument air

To control room	80 scfm
To experimenter's cell	15 scfm
Total for eight cells	120 scfm
Total to instruments	200 scfm

Plant air to each cell	50 scfm
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A communication system will be installed to provide the following services:

1. one master station for each experimenter's cell,
2. two master stations for the crane bay,
3. one master station for the power supply area,
4. one master station for motor-generator set building,
5. one master station for the fabrication shops.

Each master station is to include facilities for paging and each cell will be provided with an evacuation alarm.

Design work is under way at ORNL on the experimental crane shed, the cell personnel air lock, and the electrical system for the loops. The crane shed will house a 20-ton crane which will be used to service the experimenter's cells. The cell personnel air lock is required to provide access to a cell during operation. The layout design of the air lock is essentially complete. A preliminary single-line diagram of the electrical service to the cells has been prepared. The diagram shows two separate power supplies - one from the EGCR 13.8-kv substation and the other a 13.8-kv transmission line from the Y-12 area. The system has 6500 kva of transformer capacity to service the loop compressors,

heaters, transfer pump, and cooling water pumps. The compressor motors will be driven by individual variable-frequency motor-generator sets. Each of the main cooling water pumps will be fed from separate power sources. The standby pump can be serviced by either power source.

Experimental Fuel Handling

A study of handling problems associated with experimental fuel elements has indicated no major interferences with present fuel-handling provisions. The design of the reactor service machine has been modified to provide for servicing the through-tubes, as well as the reactor fuel and control rod channels. In order to accomplish this, it was necessary to reduce the length specification for experimental fuel assemblies to 42 ft 9 in. This provides 10 ft 3 in. of the active core depth for installation of instrumented fuel assemblies. If it is determined desirable to use the remaining 4 ft 9 in. of active core for uninstrumental fuel, the facilities required to accomplish this will have to be designed into the through-tube.

Coolant Cleanup System

Copper oxide is being evaluated as an oxidizing agent for low concentrations of H_2 and CO in helium. This material has been used as an analytical oxidizing agent, but there is not sufficient information available for design of a large-scale system. Data obtained in current studies of hydrogen oxidation have been correlated with the following design equation:

$$V = \left(\frac{F}{\pi K_1} \right) \ln \left(\frac{Y_0}{Y_0 - X'} \right) ,$$

where

V = volume of CuO bed,

F = flow rate of gas to bed,

K_1 = over-all reaction rate constant,

Y_0 = initial mole fraction of hydrogen,

X' = conversion of reactant.

It has also been established that K_1 varies with temperature by the following equation:

$$K_1 = 0.556 \exp \left(- \frac{2000}{T} \right) ,$$

where T is temperature in $^{\circ}\text{K}$.

A fission-gas collection system, consisting of a silver bed and activated charcoal, has been sized for effective holdup of the gaseous fission products that will be released from experimental fuel elements. The over-all reduction of gaseous activity in a loop operating at 1.5 Mw will be approximately a factor of 10 based on predicted release rates from the fuel and a 1% side stream flow through the traps.

Mechanical Joint Service Testing

The original objectives of this program were to evaluate a mechanical joint concept specified for the EGCR experiment loop through-tube closures and to evaluate double-gasketed gas-buffered mechanical joints for other loop applications. Since it is currently proposed to eliminate the through-tube mechanical closures by the use of remote-welding techniques, emphasis is being placed on the evaluation of mechanical joints for other loop applications.

Three test stands are currently in operation. Stand No. 1 includes an electric furnace suitable for thermal cycling small mechanical joints by applying external heating and cooling. Stand No. 2 provides for thermally cycling 10-in.-diam joints by applying internal heating and cooling. This stand is equipped with an external vacuum container for mass-spectrometer helium leak detection with the test unit at operating temperature and pressure. A diagram of this apparatus was presented previously.³ Stand No. 3, shown in Fig. 7.4, includes internal heating and has provisions for applying bending moments and axial loads with a

³"GCR Quar. Prog. Rep. Dec. 1959," ORNL-2888, p. 195.

Fig. 7.4. Apparatus for Applying Bending Moments and Axial Loads to Mechanical Joints.

hydraulic cylinder. A summary of the results of the tests that have been conducted in these facilities is presented in Table 7.7.

Two types of mechanical joints have shown promise for high-temperature service with helium. These are the Conoseal joint manufactured by the Marman Division of Aeroquip Corporation and the Grayloc joint manufactured by the Gray Tool Company. A 2 1/2-in. gas-buffered Marman Conoseal joint is shown in Fig. 7.5, and a 2 1/2-in. standard Grayloc joint is shown in Fig. 7.6. Five-inch gas-buffered Marman joints and 6-in. gas-buffered Grayloc joints are currently on order for additional proof tests.

In addition to the Marman Conoseal and Grayloc joints, preliminary room-temperature tests have been conducted on gas-buffered joints manufactured by the DSD Manufacturing Company and the Hydrodyne Corporation. These joints have shown room temperature helium leak rates in excess of the specified maximum allowable leakage for EGCR loop application of

Table 7.7. Summary of Results of Elevated-Temperature Tests of Mechanical Joints

Type of Test	Type of Joint	Test Conditions	Results
Screening tests in stand No. 1	2 1/2-in. standard Marman Conoseal	111 thermal cycles between room temperature and 500°F 106 thermal cycles between 500 and 1000°F 35 thermal cycles between 700 and 1200°F Internal pressure, 375 psig	The joint had a maximum helium leakage rate of 5.9×10^{-10} cm ³ (STP)/sec until completion of 23 thermal cycles between 700 and 1200°F, at which time the leakage increased to $>2 \times 10^{-4}$ cm ³ (STP)/sec; the joint was remade with a new gasket and again leaked after 12 cycles between 700 and 1200°F
	2 1/2-in. standard Marman Conoseal	11 thermal cycles between 600 and 1100°F	The joint leaked on the 11th cycle
	2 1/2-in. standard Grayloc	103 thermal cycles between room temperature and 500°F 125 thermal cycles between 500 and 1000°F Internal pressure, 375 psig	The joint remained leaktight to beyond the sensitivity of the helium leak detector (1.1×10^{-10} cm ³ (STP)/sec throughout this test, including a final room-temperature check; however, the cadmium plating on the sealing ring melted during operation at 1000°F
	2 1/2-in. standard Grayloc with type 410 stainless steel seal ring	1 thermal cycle between 500 and 1000°F Internal pressure, 375 psig	The joint leaked after the first complete thermal cycle
	2 1/2-in. gas-buffered Marman Conoseal	56 thermal cycles between 200 and 700°F with an internal pressure of 400 psig; test continuing	Leakage across the inner seal is $<1.1 \times 10^{-10}$ cm ³ (STP)/sec; leakage across outer seal is 2.6×10^{-7} cm ³ (STP)/sec
	10-in. standard Marman Conoseal	61 thermal cycles between room temperature and 500°F with an internal pressure of 400 psig; test continuing	Maximum leakage rate is $\sim 1 \times 10^{-5}$ cm ³ (STP)/sec; leakage rate after 61 cycles with the joint at 100°F and the internal pressure reduced to 20 psi was 8.4×10^{-7} cm ³ (STP)/sec
Bending-moment tests in stand No. 3	10-in. standard Marman Conoseal	50 bending-moment cycles between 0 and 290 000 in.-lb with the joint at room temperature 50 bending-moment cycles between 0 and 240 000 in.-lb with the joint at 500°F 50 bending-moment cycles between 0 and 240 000 in.-lb with the joint at 1000°F 50 axial compression load cycles between 0 and an equivalent of 48 000 lb compression at 1000°F; test continuing	Maximum leakage rate during these tests was 1.8×10^{-6} cm ³ (STP)/sec

Fig. 7.5. Gas-Buffered Marman Conoseal Mechanical Joint for 2 1/2-in. Pipe.

$1 \times 10^{-6} \text{ cm}^3 \text{ (STP)/sec.}$ Therefore only the Marman Conoseal and Grayloc joints have been tested at high temperatures.

The 10-in. Marman Conoseal joints failed to provide satisfactory sealing with the gaskets originally furnished. The manufacturer indicated that thicker gaskets should have been provided, so he furnished replacement gaskets. The new gaskets substantially improved the sealing. Leaktight closures were obtained at room temperature, but, at operating temperature and pressure, leakage was slightly over the maximum allowable leakage. A dimensional check disclosed that the flanges had warped during welding into the test fixture and that the sealing surface was as much as 0.010 in. out of round, in some places. Such dimensional changes may account for the large joint not being as leaktight as the smaller joints. Special precautions will have to be taken to prevent flange warpage during welding of the larger joints.

While the first thermal cycling test of a Grayloc joint was entirely successful, the test piece utilized a cadmium-plated gasket that would

Fig. 7.6. Standard Grayloc Mechanical Joint for 2 1/2-in. Pipe.

be unsuitable for high-temperature reactor applications. A second Grayloc joint with a type 410 stainless steel unplated sealing ring leaked when cooled to 500°F after a period of operation at 1000°F. A third joint with a nickel-plated sealing ring also failed to seal. The flanges of these joints were carbon steel. A final evaluation of this type of joint for high-temperature service cannot be made until all-stainless-steel joints are available for testing.

Special Compressors

Grease-Lubricated Centrifugal Compressors

The grease-lubricated centrifugal compressor has satisfactorily completed a total of 3500 hr at 12 000 rpm with helium at 590°F and 400 psia (suction condition). With the exception of repairs to the

gasoline-driven motor-generator sets, the total operating time was accumulated without extended shutdowns. The compressor was disassembled for postoperation inspection, and it was found that the rear bearing (motor end) and lubricant were in good condition. The front bearing (impeller end) was in a satisfactory condition, but the grease had become discolored and its lubricity had deteriorated. The inside diameter of the front bearing housing had increased from 0.002 to 0.0025 in., and the housing surface was slightly tapered. The stator-fit diameter had increased approximately 0.004 in., and a slight ovality was noted. It was also found that the coolant flow through the bottom water-spiral plate and cold finger assembly had been inadequate. The compressor housing has been modified to accommodate the regenerative impeller and casings, and it will be used as one of the compressors for GCR-ORR loop No. 1.

Regenerative Compressors

Assembly of the GCR-ORR loop No. 1 compressors, fabricated by A-Tool and Gage Company, has been delayed because of unsatisfactory water flow distribution through the coolant passages. The coolant flow provides the heat sink necessary to maintain operable conditions for the grease-lubricated bearings. The first unit assembled failed, as mentioned previously,⁴ during proof-testing. The initial inspection disclosed a faulty bearing (14 balls instead of the 15 required), and further inspection and tests revealed unsatisfactory coolant circulation through the main housing coolant passages and the cold finger as a result of too small a resistance to flow across the assembly. Two schemes for solving the coolant flow problem are being investigated simultaneously. One scheme consists of electroless nickel plating (Kanigen process) of the spiral passageways. This is being undertaken by the General American Transportation Corporation. Geometry may be the limiting factor in the success of this scheme. The other scheme consists of replacing the outer shell assembly to provide for a shrink fit between the shell and the

⁴"GCR Quar. Prog. Rep. March 31, 1960," ORNL-2929, p. 251.

spiral drum. Misalignment of bearing fits, as a result of welding, may limit the success of this mechanical repair scheme. The immediate requirement for two regenerative compressors for GCR-ORR loop No. 1 is being filled by utilizing the prototype regenerative compressor housing and by modifying a centrifugal compressor housing, as mentioned in the preceding section, to suit the geometry of the regenerative impeller and casings. The flow of water through the passages in the main housing was satisfactory for these particular units. A method for improving the cold finger assemblies has been devised. The compressor housings which are being modified as described above will be assembled into spare units.

Compressors With Gas-Lubricated Bearings

A new journal and bearing unit, with a diameter of 1.5 in., a diametral clearance of 0.009 in., and a length of 1.5 in., was obtained for testing. A second unit having a diameter of 2 in., a diametral clearance of 0.0013 in., and a length of 2.5 in. has been ordered in an effort to find a reliable source of precision machining required for these components. No actual testing was performed during the quarter because of inability to regulate the speed of the generators, which are driven by an internal combustion engine. A new, electric-motor-driven generator set with a magnetic clutch has been ordered and is to be installed at the beginning of the next quarter.

The fabrication of the facility for testing the Bristol-Siddeley compressor was completed with the aid of a mockup of the compressor suction and discharge flanges. No further progress is practicable without the compressor. Bristol-Siddeley has reported difficulty with the test-loop throttling valve and inability to obtain cast Monel of the quality required for the bearings.

High-Temperature Gas-Bearing-Compressor Test System

The gas control cabinet, motor control cabinet, and the variac cabinet have been fabricated and installed in the system for testing gas-bearing compressors at high temperature. The test facility is complete except for the compressor and the throttling valve.

Some ceramic insulation broke loose from the compressor motor stator during fabrication and exposed part of the windings. It was necessary to return the stator to the Louis-Allis Company for repair. The repairs were made satisfactorily, according to Louis-Allis engineers, and the stator was returned to A-Tool and Gage Company, the compressor motor vendor.

During the final grinding of the rear journal, two areas having very small pit marks were uncovered. It has not yet been determined whether the entire shaft will have to be remade. It does not appear from the progress to date that a definite delivery time can now be established.

Compressors for GCR-ORR Loop No. 2

A contract was awarded to Bristol-Siddeley Engines, Ltd., to supply three gas-bearing compressors, with an option to purchase one additional compressor. Delivery of two compressors was promised in December 1960, with delivery of the third (spare) compressor in April 1961. The compressor casing will be made of type 317 stainless steel instead of type 347, as originally specified, to avoid a ten-week delay associated with obtaining type 347 stainless steel in England.

The order was placed on the basis of acceptance of a hydrogen-palladium leak test rather than the mass spectrometer test specified. Bristol-Siddeley has subsequently agreed to the more stringent mass spectrometer leak test.

Compressors for EGCR Experiment Loops

Visits were made to each compressor manufacturer that had been invited to submit proposals for the gas compressors for the EGCR in-pile loops in order to evaluate their facilities and capabilities. Several firms indicated that it would be difficult to design one motor that would meet the low-speed, high-horsepower requirements for normal loop operation with carbon dioxide and emergency operation (two of three compressors running) with helium. An addendum to the specification was

issued to permit the compressor manufacturers to obtain the required gas mass flow and original test conditions for the two helium conditions by increasing the system gas pressure and, thus, the gas density, in inverse proportion to the decrease in speed. The closing date for acceptance of bids was extended one week.

Formal proposals to supply the gas compressors were received from four firms on March 29, 1960. A contract was awarded to Bristol-Siddeley to supply 16 gas-bearing compressors, with an option to purchase seven additional compressors. Delivery of the first set of three compressors was promised for July 15, 1961.

Study of Back-Diffusion Through Labyrinth Seals

Data on back-diffusion through labyrinth seals have been obtained under the following initial test conditions:

Gas	
Pressure, psig	100
Temperature, °F	100 to 600
Buffer water	
Pressure, psig	100
Temperature, °F	90 to 100
Seal exhaust	
Pressure, psig	90 to 99
Temperature, °F	Up to 195
Shaft speed (constant), rpm	3600
Labyrinth radial clearance at room temperature, in.	0.003

The radial clearance of the labyrinth decreases as the operating temperature increases. There was no detectable back-diffusion of water into the gas stream at the above test conditions.

The tests presently in progress are being conducted at higher pressures and temperatures. These tests with various differential pressures should establish the limits at which back-diffusion occurs.

Fig. 7.7. Test System for Studying Back-Diffusion Through Labyrinth Seals.

The general labyrinth arrangement, the flow paths of the gas, buffer water, seal exhaust, and the back-diffusion detection location are shown in Fig. 7.7.

Previous reports in this series are:

ORNL-2500 Parts 1-4, issued April 1, 1958
ORNL-2505 Issued June 19, 1958
ORNL-2510 Issued September 18, 1958
ORNL-2676 Period Ending December 31, 1958
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