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CONF-760530--1

Design, Operation, and Initial Results from a Series of Graphite Creep Irradiation Experiments*

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Abstract

A series of irradiation tests was designed to evaluate the creep characteristics at elevated temperatures and to high fast fluences of various graphites of interest to HTGR designers. The series encompasses the irradiation of 28 specimens 15.24 mm (0.6 in.) diam × 25.4 mm (1 in.) long at 900°C to incremental exposures of 1, 2, 4, and 8 × 10^{21} neutrons cm^{-2} (E > 0.18 MeV); 28 similar specimens at 600°C to the same exposures; and 28 other similar specimens at 1250°C under the same conditions. A compressive stress of 13.79 MPa (2000 psi) is applied to 20 of the specimens in each test by means of a metal bellows expanded by gas pressure against the specimen columns. Eight of the stacked specimens in each test are stressed to 20.68 MPa (3000 psi) by a reduction in diameter. Special features of the capsules are described which include (1) moveable center-line thermocouples which measure the temperature profile along the axis of the capsule, (2) linear variable differential transformer-type load cells to monitor the applied load, and (3) computerized temperature control designed to achieve accurate longitudinal temperatures over the 0.508 m (20 in.) length of the test specimen columns. Results achieved from irradiation of the first capsule, one of a total of twelve in the series to be irradiated over a four-year period, are presented.

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*Research sponsored by the U.S. Energy Research and Development Administration under contract with the Union Carbide Corporation.

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Introduction

The fast-neutron dimensional distortion produced in graphite can produce high internal stresses in graphite. In general, these stresses do not become severe due to fast-neutron radiation-induced creep which provides a stress relaxation mechanism. It is apparent that, for the proper design for safety and operations of reactors that use graphite in their cores, the creep coefficients must be well known. This is necessary not only to calculate anticipated static stress loads in regions of the core where thermal and fluence gradients occur, but also to evaluate abnormal transient conditions such as thermal stress on shutdown and startup and superimposed cyclic loading. Data exist for a number of graphites of the United States, European, and Russian origin. These data are scattered — over a factor of 3 to 5 — and the graphites do not correspond to the coarse-grained isotropic graphite grades being considered for large scale high-temperature gas-cooled reactors (HTGRs). It is obviously necessary to develop such creep data for these materials. The work as described below is a part of the overall programs by General Atomic Company (GAC) and the Oak Ridge National Laboratory (ORNL) for the characterization and standardization of graphites employed in HTGRs.

Our basic objective in this is to determine the primary and secondary creep coefficients for the neutron-induced creep of these graphites at 600, 900, and 1250°C for accumulated fluences $\geq 8 \times 10^{21}$ neutrons/cm^{-2} (E > 0.18 MeV).‡

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†Speaker.

‡Unless otherwise stated, all fast-neutron energies shall be of this value.

The maximum design fluence accumulation for core graphite of a 1000 MW(e) central station HTGR during the reactor lifetime is 8×10^{21} neutrons/cm^{-2}.

Program Objectives and Design Criteria

Principal Objectives

Our program plan for the study of the fast-neutron-induced creep of graphite consists of a series of 12 irradiation capsules, OC-series, for which the source of fast neutrons will be the Oak Ridge Research Reactor (ORR). The program has two main objectives. These are to (1) design and construct a flux-trapped irradiation facility in the ORR E-5 position, and (2) determine both the primary and secondary creep coefficients and other property data required for the constitutive equations for graphites employed in high-temperature reactors.

Type of Test

The best way to accurately determine the creep coefficients for any set of conditions would be to continuously monitor the creep strain during the test; however, the limitations on the quantity of specimens that could be tested, the complications involved in the test design and the maintenance of the integrity of strain gages or dilatometers make this impractical at this time for tensile or compressive tests. We selected the simplest test — a constant stress compressive test — as the basis for our design. This type of test is less complex and, therefore, less expensive than tension stress tests. It has the additional advantage that the secondary creep strains are reported[1] to be approximately equal for compression and tension stresses.

Test Materials

The selection of the first materials for these tests was predominantly influenced by the current reactors in operation and/or design. The core material, graphite grade H-327, for the Fort St. Vrain reactor is represented by sufficient numbers of specimens to substantiate its anticipated creep behavior. A small number of specimens of graphite grade AXF-8QBG1 previously irradiated

at the Battelle Pacific Northwest Laboratories to 1.0×10^{22} neutrons/cm^{-2} (E > 0.18 MeV) are included for the purposes of extending data on creep behavior to very high fluences after breakaway expansion. The major portion of the specimens in these tests is from grade H-451 graphite. This is a near-isotropic material manufactured by the Great Lakes Carbon Corporation and is one of the principal contenders for 1000 MW(e) HTGR application. It was selected as the major material to start this series because it, at this time, is the most thoroughly characterized material of its kind. It appears to be an acceptable material.

Test Parameters

In our basic objective, the primary and secondary creep coefficients will be determined as functions of fluence, temperature, stress, and Young's moduli of elasticity. These will consist of a series of irradiation capsules that are alternately irradiated, in the order of priority, at 900, 600, and 1250°C in fluence increments of approximately 1.0×10^{21} neutrons/cm^{-2}(E > 0.18 MeV) until each set of test specimens for each test temperature has accumulated a fluence in excess of 8×10^{21} neutrons/cm^{-2}, the full design fluence for a full-scale HTGR. The compressive stresses at each temperature will be 2000 psi (13.8 MPa) and 3000 psi (20.7 MPa). The dimensions, Young's modulus, and shear modulus for each graphite specimen will be determined prior to neutron irradiation and subsequently after each accumulated increment of fluence, approximately 1.0×10^{21} neutrons/cm^{-2}, one cycle in the ORR. As explained later in more detail, each capsule will contain a set of unstressed specimens that match the stressed specimens. These unstressed specimens serve as control specimens and are characterized before and after irradiations as are the stressed specimens. Basically, the total creep strains are determined by the dimensional differences between stressed specimens and their matching control specimens after each cycle in the ORR. The primary creep strain has been found to be recoverable under irradiation if the stress is removed.[2] Therefore, selected stress-tested specimens will be irradiated again at their test temperature to recover and thereby

determine their primary creep strains. Our objective is to acquire the creep data to an accuracy within 10%; that is, reasonably good engineering design data are sought.

Design of the ORR Facility and the 900°C Creep Capsule

Flux-Trap Irradiation Facility

As indicated, the capsules for each experiment will be operated sequentially in the ORR E-5 core facility. This requires only a minor rearrangement of the ORR core loading pattern to permit a flux-trap irradiation facility to be constructed. This location within the ORR core and the general configuration of an experiment are shown in Figs. 1 and 2. The E-5 core position provides a large 3 × 3 in. (76 × 76 mm) water-cooled facility that will accommodate an experiment of up to about 2.8 in. (71 mm) in overall diameter. The experiments will be placed in the E-5 position inside an aluminum core filler piece.

Capsule and Test Specimens

Each experiment is contained inside a type 304 or 304L stainless steel container. Basically, an experiment consists of a high-density isotropic graphite* holder that is 2.347 in. (59.61 mm) diam × 23¼ in. (591 mm) long with four longitudinal holes 90 deg apart with each hole containing 14 diametrically centered graphite test specimens, thermal trimming heaters, thermocouples, and flux monitors. However, beyond this, the accuracy and precision required plus the complexity of this most direct approach require complex designs and the introduction of sophisticated equipment into the design of the capsules and the irradiation test facility.

Capsule OC-1 is designed to irradiate twenty-eight 0.6000 in. (15.24 mm) diam by 1.0000 in. (25.4 mm) long compressively stressed graphite test specimens at 900°C to the first planned exposure level of 1×10^{21} neutrons/cm^{-2}.

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*Grade AXF-8Q manufactured by POCO Graphite, Inc., a subsidiary of Union Oil Corporation, Decatur, Texas.

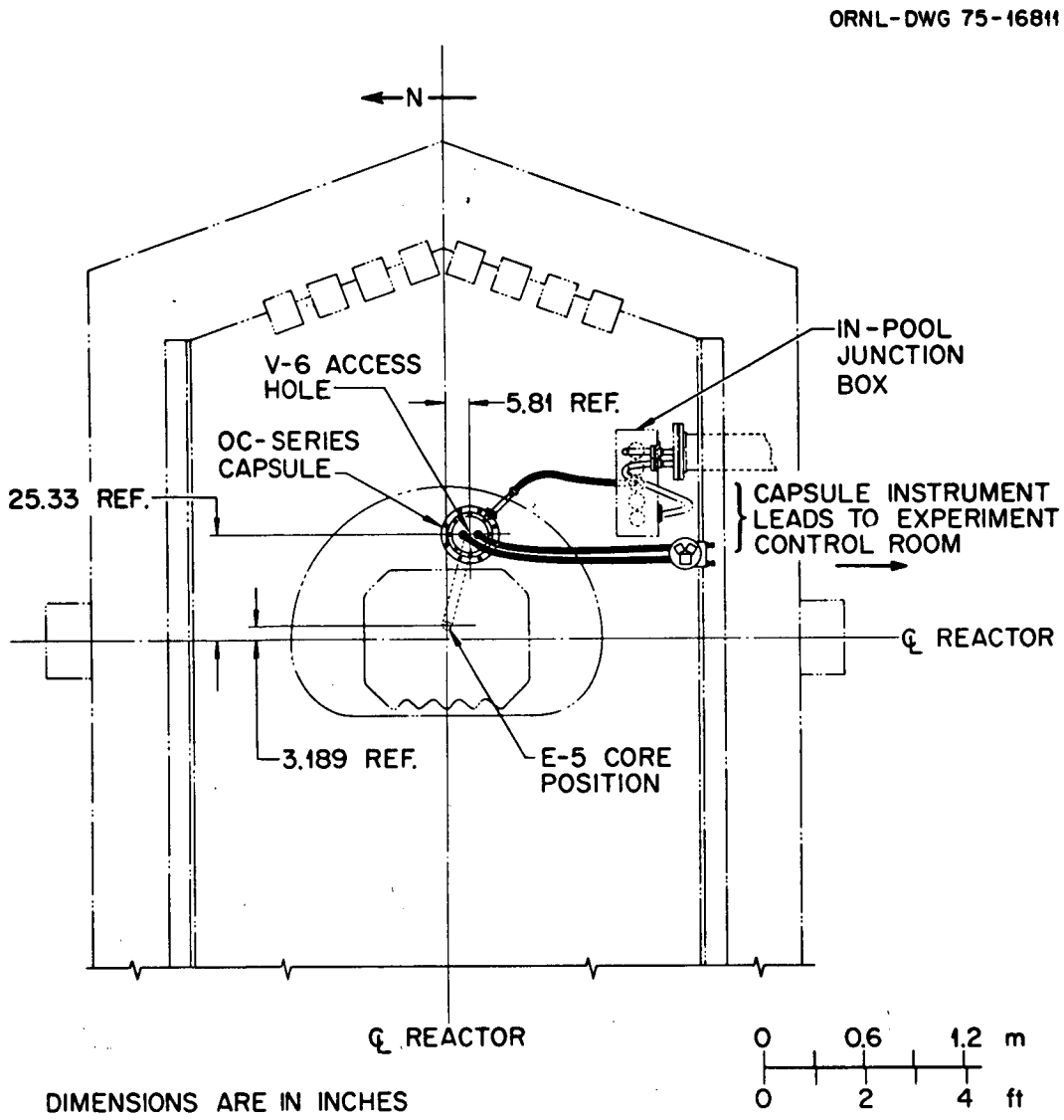

Fig. 1. A Top View of an OC-Series Capsule Installed in the ORR E-5 Position.

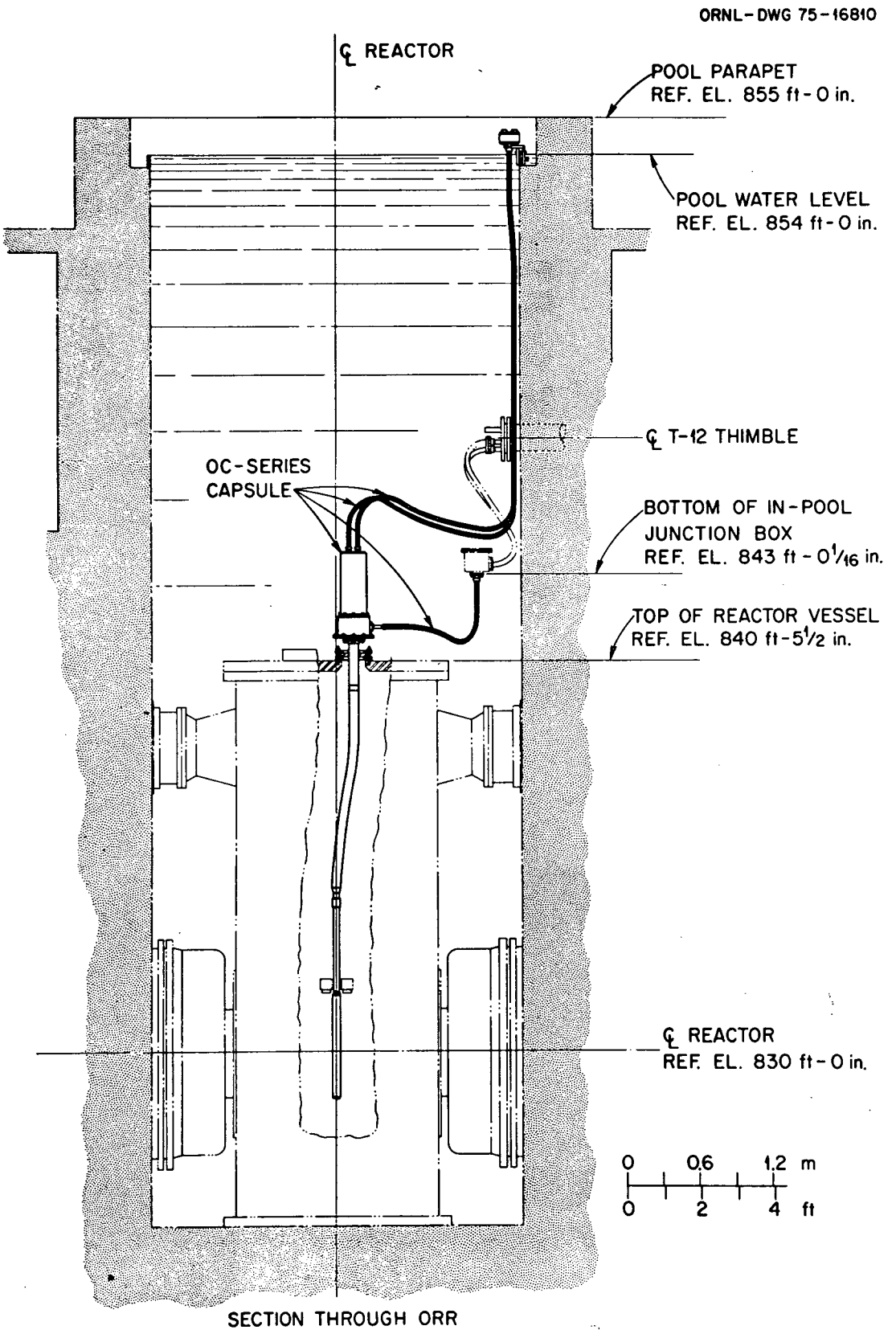

Fig. 2. An Elevation View of an OC-Series Capsule Installed in the ORR E-5 Position.

A compressive stress of 2000 psi (13.8 MPa) is applied to 20 of the specimens in the test by precalibrated metal bellows expanded by gas pressure against the specimen columns. Eight of the specimens in the two columns have reduced diameters so as to increase the stress to 3000 psi (20.7 MPa). Each capsule also includes 28 unstressed control specimens made of the same types of graphite as are the stressed specimens.

Special features of the capsule design include (1) movable center-line thermocouples which measure the temperature profile along the axes of the control specimens of the capsule, (2) special linear variable differential transformer (LVDT) type load cells to monitor the applied load, and (3) computerized temperature control designed to achieve accurate and precise uniform longitudinal temperatures over the 20 in. (0.508 m) length of the test specimen columns. These features are described in more detail below.

Cross-sectional views of the in-core portion of the capsule are shown in Figs. 3 and 4. Figure 3 shows the two stressed specimen columns. The specimens each have shallow holes in each end to accommodate the graphite pins which pass through the spacers to serve as centering guides for the specimen column. The spacers center the column by means of longitudinal centering ridges protruding from their cylindrical surfaces. This centering is important to the proper loading and temperature control of the specimens. Each metal bellows, shown at the top of the two specimen columns, will be pressurized with helium gas to 854 psig (4.89 MPa) to provide a load of 565.4 lb (2515 N) which gives the required compressive stress. Force from each bellows is transmitted to the specimen column through a graphite push-rod. At the end of each specimen column, the force is sensed by an LVDT-type load cell. This device is designed to measure up to 700 lb (3115 N) at 480°C. Although monitoring of the pressure in the bellows will be the primary measure of the load being applied to the specimen columns, the LVDT-type load cell will provide backup information during the early stages of the irradiation and ensure that the force is indeed transmitted through all of the specimen column. The load cell is expected to suffer

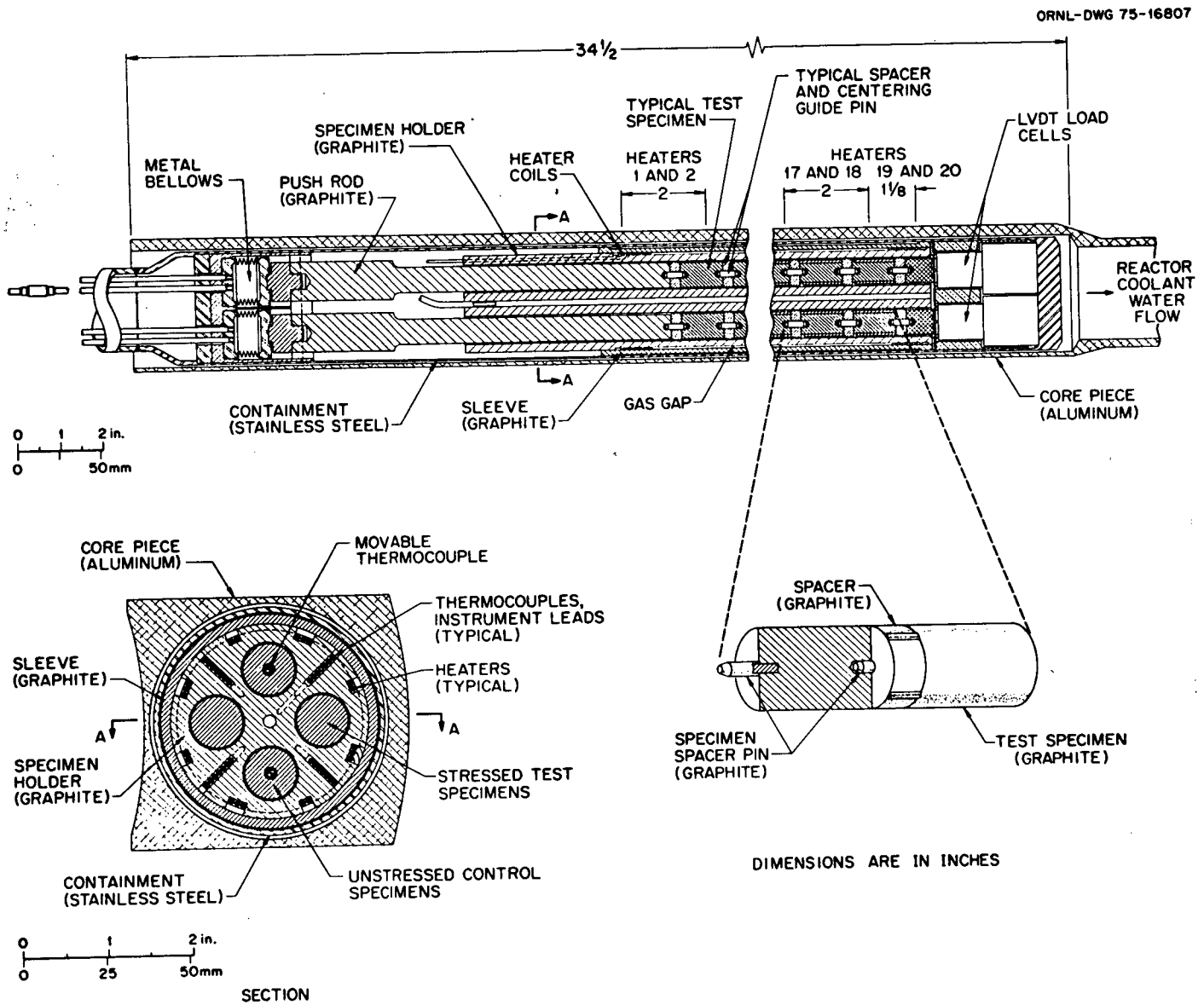

Fig. 3. A Cross-Sectional View of the In-Core Portion of the ORR Capsule OC-1 Showing the Stressed Columns.

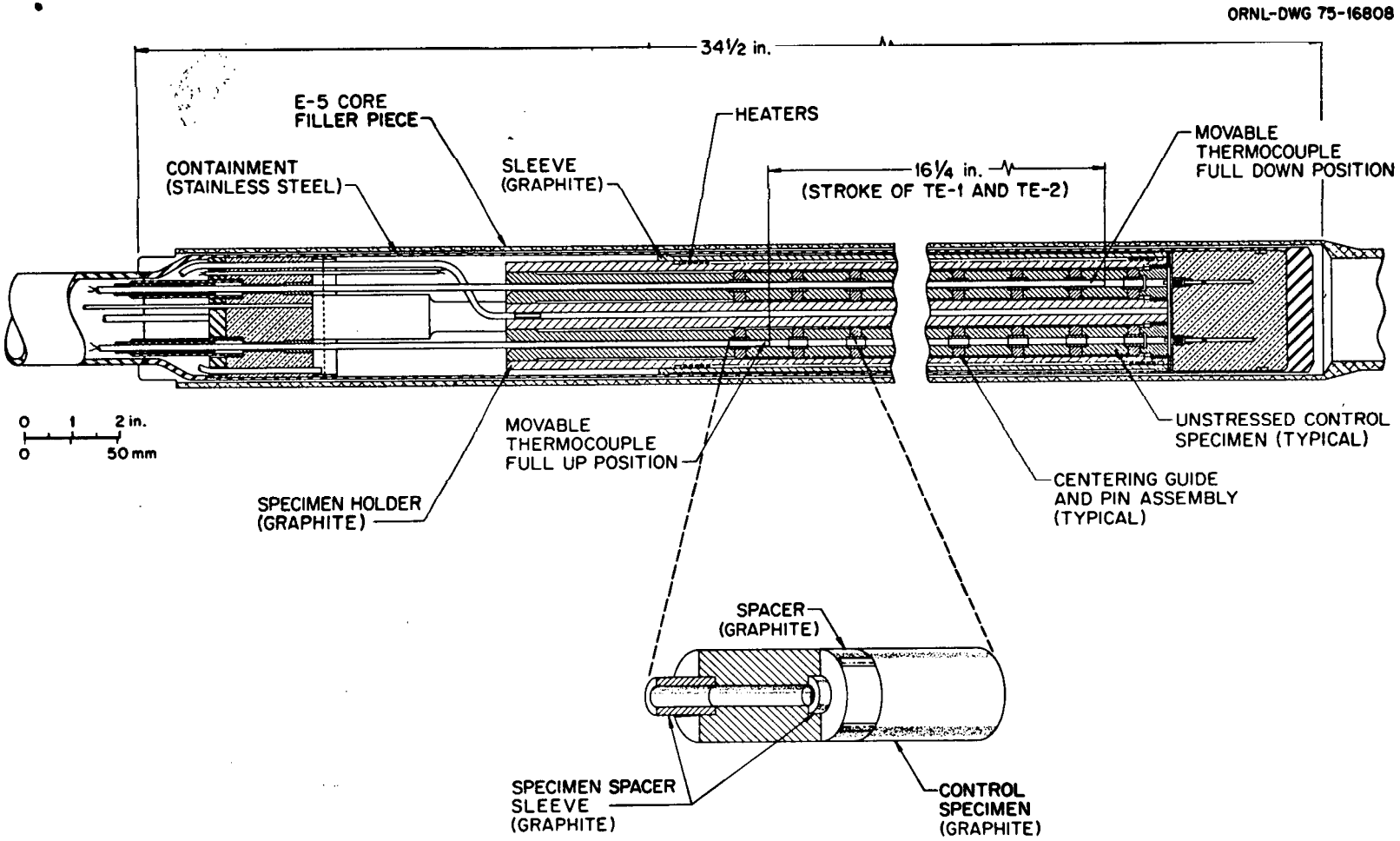

Fig. 4. A Cross-Sectional View of the In-Core Portion of the ORR Capsule OC-1 Showing the Unstressed Columns.

radiation damage and decalibration at exposures exceeding 3×10^{20} neutrons/cm^{-2} (E > 0.18 MeV), but it will provide valuable information during the initial stages of capsule operation.

The unstressed control specimen columns are shown in the cross-sectional view in Fig. 4. These specimens are stacked and centered in a manner similar to the stressed specimens, except that a longitudinal hole has been provided to serve as a guide for the 1/8 in. (3.2 mm) diam stainless-steel-sheathed Chromel-Alumel movable thermocouple that is installed in each column. These thermocouples can be moved through a 16¼ in. (0.413 m) stroke, thus providing temperature traverses from the midplane of the uppermost specimen to the midplane of the lowest specimen in each column. The sheathed thermocouples pass up through the various bulkheads and through a 1/4 in. (6.4 mm) diam guide tube to the junction box located just above the reactor vessel top flange. This junction box houses rack-and-pinion assemblies which drive the thermocouples. A rack is attached to the top end of each movable thermocouple. An electrically driven motor drives the rack-and-pinion gear assembly and the attached thermocouple up and down at a rate of 2 in./min (0.83 mm/s). A variable resistor driven by a gear from the rack provides a signal related to the position of the thermocouple. By operation of the motor, the thermocouple position can be controlled to less than ± 1/8 in. (± 3.2 mm) in longitudinal elevation. The thermocouple signal is transmitted to lead wire by means of sliding contacts which move along a gold-plated insulated strip which serves as the conduit from the contacts to the lead wire. Bench tests have demonstrated that the units are mechanically reliable and that no detectable error is introduced in the thermocouple temperature signal.

In addition to the two movable thermocouples, twenty-six 1/16 in. (1.6 mm) diam stainless-steel-sheathed MgO-insulated Chromel-Alumel thermocouples have been provided to monitor capsule temperatures and/or for control purposes. Of these, 23 are located within the graphite specimen holder adjacent to the test specimens at various elevations along the capsule.

Temperature Control of Specimens

As stated previously, one of the important objectives of the experiment is to maintain accurate and precise temperature control along the specimen column.

To achieve adequate control, the capsule is designed to operate on gamma heat alone approximately 100°C below design temperature. The gamma heat rate in this experiment position in the reactor varies from a low of about 5.4 W/g to a high of about 10 W/g, as shown in Fig. 5. The design is for a thermal resistance helium-filled gas gap between the specimen holder sleeve (see Fig. 3) and the water-cooled capsule vessel stepped in a manner that provides five different radial gaps. These gaps are designed to accommodate the variation in gamma heating rate and to establish approximately the desired constant longitudinal temperature level.

Design operating temperature and control are achieved by twenty 1/16 in. (1.6 mm) diam stainless-steel-sheathed MgO-insulated Nichrome V heaters wrapped around the specimen holder as shown in Figs. 3 and 4, and retained there by the graphite sleeve that is slipped over the holder. Ten 2-in.-long (50-mm-long) heated zones, each with two heaters operated in parallel, are positioned along the 20 in. (0.51 m) length of the specimen holder. Each heater is capable of operation at up to 50 W/linear inch (20 W/lineal cm); however, they are expected to operate well below this maximum in order to maintain the design operating temperature of 900°C for the specimens.

The HEATING3, described in ORNL-TM-3208,[3] was used for all heat transfer calculations. HEATING3 is a finite difference code stored for easy accessibility in the library for computers at ORNL. This program has been in use at ORNL for several years and has proved satisfactory. It (with appropriate modeling) was used to determine the dimensions of the five different radial gaps used in the graphite sleeve to accommodate the variation in the gamma heating rate and to make steady-state, transient, and fine nodal modeling heat-transfer calculations; also it was used in the development of the mathematical relationship for the computer, described below, to calculate the center-line temperatures of the test specimens relative to the outer gas control gap depth in mils (the radial gaps on the outside of the graphite sleeve on the specimen holder), longitudinal axial position in inches, and heater power in watts per lineal inch. Calculations indicated that the greatest heater power requirements to trim the specimen

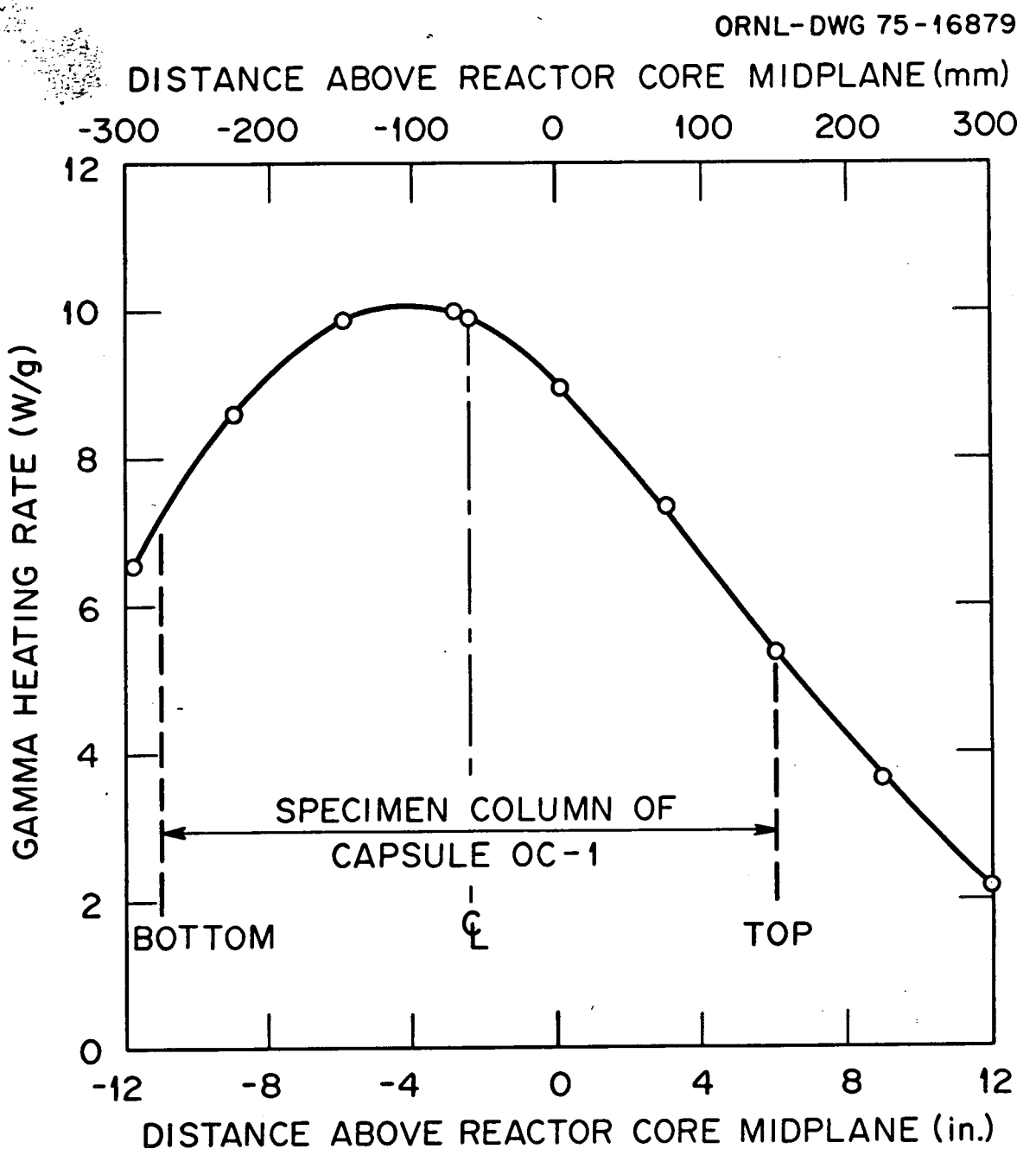

Fig. 5. Results of Gamma Heating Measurements Made in the E-5 Core Facility of the ORR on September 19, 1974.

temperature to the desired 900°C would be for those heaters included in the third zone down from the top down to but not including the bottom heater zone. This was expected to be approximately 11.4 W/lineal inch of heater, less than 23% of the maximum rated power of the heaters.

Computer and Ancillary Equipment

Because of a requirement to maintain a ±10°C temperature control* along the specimen column and the intricate interaction of each heater zone with respect to adjacent heater zones, a computer was acquired to control the experiment temperatures automatically. A Digital Equipment Corporation PDP 11/40 computer with the required ancillary equipment was procured to operate the capsule heaters and to monitor and record the data from the experiment. Algorithms have been developed which relate the various thermocouple temperatures to the specimen temperatures and thus permit the computer to adjust the heaters to the required operating levels. Ten solid-state heater control devices are used (one for each zone) which operate under computer control as long as the computer is operating. In the event of loss of signal from the computer, the controller will sense the latest temperature from its control thermocouple and continue to control the heater output to maintain this temperature. Computer control is resumed as soon as it is operating again.

Test Specimens Configuration and Testing Plans

The tentative arrangement of the specimens in the loaded columns of OC-1, the start of the 900°C series of experiments, is outlined in Table 1. As previously stated, the grade of material and positions of each loaded specimen is matched by control specimens in the unloaded columns (see Figs. 3 and 4).

The loading of the specimens in the OC-2 experiment, the start of the 600°C series of experiments, will be like that in OC-1. In the OC-3 experiment, the start of the 1250°C series of experiments, the plan is to use the same type of loading configuration used for OC-1 and -2 except that specimens from grade H-451 shall be used in the positions labeled as AXF-8QBG1 in Table 1.

As previously stated, irradiation data have indicated that the primary creep is recoverable if the irradiation creep-tested specimen is exposed in

*This corresponds to a ±3% change in apparent creep coefficient.

Table 1. Configuration of the Compressively Stressed Specimens in Columns *E* and *W* of Capsule OC-1 to be Operated at 900°C

TOP

		Column "E"			Column "W"		
Position	Grade[a]	Orientation	Stress[b]	Modulus	Orientation	Stress[b]	Modulus
1	H-451	‖			‖		
2	H-327	‖			‖		
3	H-451	‖			‖		
4	H-451	‖	High		‖	High	
5	H-451	‖			‖		
6	H-451	‖		Low	‖		Low
7	H-451	‖		High	‖		High
8	H-451	‖		Inter-mediate	‖		Inter-mediate
9	H-451	Radial			Radial		
10	H-451	‖	High		‖	High	
11	H-451	‖			‖		
12	H-451	‖	High		‖	High	
13	H-327	⊥			⊥		
14	AXF-8QBG1	Isometric	High		Isometric		

BOTTOM

[a]The stock for graphite grades H-327 and H-451 are in the form of right circular cylinders. The symbol ‖ means that the axis of rotation of the specimen is parallel with that of the stock and *with grain;* the *radial* orientation means that the axis of rotation of the specimen is parallel with a radius of the stock and *across grain.*

[b]The stress is 2000 psi (13.79 MPa) except for those labeled *high* which indicates that the stress is 3000 psi (20.68 MPa).

the unloaded condition to fast neutrons at the previous irradiation temperature. Therefore, to acquire the necessary data to calculate the primary creep coefficients, selected specimens will be removed from the loaded columns and irradiated again in an unloaded column of specimens. This will start with the OC-4 experiment when the set of specimens from OC-1 are returned in OC-4 for their second incremental accumulation of fast fluence. The plan is illustrated

in Table 2.* Two of the compressively creeped specimens from the OC-1 experiment are loaded into the unloaded columns of the OC-4 experiment, which are indicated in the row marked *Primary* in Table 2. The positions that these left vacant in the loaded columns of OC-4 will be filled by new, unirradiated specimens of grade H-451 (or another grade of graphite, if warranted). This technique has the effect of making a small sacrifice in the quantity of specimens that are ultimately available for analyzing for creep coefficients, but it does save considerable expense in building a separate irradiation facility to acquire the primary creep data.

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*The plan involves only specimens of graphite grade H-451 having an intermediate Young's modulus.

Table 2. Typical Plan for the Irradiation at 900°C for One Column Section of Graphite Grade H-451 Compressively Stressed Specimens

PLAN

Capsule No. →	OC-1	OC-4	OC-6	OC-8
Fluence → (neutrons/cm^2) x 10^{-21}	1	1	2	4
	Number of Specimens x Fluence			
Low Modulus	2 x 1	2 x 2	2 x 4	2 x 8
High Modulus	2 x 1	2 x 2	2 x 4	2 x 8
Intermediate Modulus	6 x 1	4 x 2	2 x 4	2 x 8
		2 x 1	2 x 3	4 x 4*
			2 x 2*	2 x 6*
Primary		2 x 1	2 x 2	2 x 3

RESULT

Number of Specimens x Fluence	
For Secondary Creep	For Primary Creep
8 x 1*	2 x 1
6 x 2*	2 x 2
2 x 3	2 x 3
6 x 4*	
2 x 6*	
2 x 8	

*Could use to introduce second graphite type.

Table 2 further illustrates the plan and results anticipated as the OC-series of experiments are conducted beyond OC-4 through the desired twelve experiments.

The irradiation schedule for each set of specimens per temperature series involves four capsules that are exposed in their turn to accumulate fluences of approximately 1, 1, 2, and 4 $\times 10^{21}$ neutrons/cm^{-2} for the total accumulation of the $\geq 8 \times 10^{21}$ neutrons/cm^{-2}. This means that each set of specimens will require fifteen months total irradiation time to acquire the desired fluence.

Operation and Initial Results

The irradiation of the OC-1 experiment was started on April 1, 1976. We have had difficulty in maintaining the 900 $\pm$ 10°C uniform temperature along the length of the specimen columns. There are two major reasons for this. The ORR core loading has included some partially spent fuel elements which has skewed the flux patterns for the gamma and fast neutron radiations. The gamma heating rate failed to bring the specimen temperatures to within the 100°C of the desired operating temperature of 900°C. Consequently, we have had to use helium-argon sweep gas instead of just helium in the capsule and to use more heater power than we had originally anticipated in order to bring the test specimen temperatures to the test temperature.

The instrumentation of the capsule has generally been a satisfying part of the experiment for the operational information that it supplies. For example, the two movable thermocouples that move up or down through the central axes of the unstressed control specimens have proven to be invaluable as aids in guiding our operations of the controls toward achieving the desired operating conditions.

Applications and Future Plans

Beyond storage of archival samples, no provision is now being made to determine primary creep and secondary creep under tension. A decision to do additional experiments toward the measurements of these coefficients shall

depend upon the degree of accuracy and precision obtained in these compressive experiments, the quality and applicability of the tension creep determinations currently being made in Europe, and the criticality of the values for core stresses and analyses.

As indicated, these experiments are primarily and currently directed toward the immediate needs of operational HTGRs and those that are being designed. As these series of tests are designed and with the material selected for testing, the data generated should also be useful to other reactor designs such as the Molten Salt (breeder) and the Controlled Thermonuclear (fusion) Reactors. As indicated above, test sites may become available for other specific grades of graphite as the series of tests progresses.

Acknowledgments

The authors are indebted to a number of people for assisting in the design, fabrication, operation of the experiment, and the preparation of this report: J. M. Jansen, Jr. and J. A. McEvers of the Instrumentation and Controls Division for procurement and assembly of the computer hardware plus the development of the software and scan routines; J. W. Cunningham of the Instrumentation and Controls Division for the development, procurement, and installation of the circuits and instrumentation for monitoring and controlling the experiment; H. C. Roland, consultant from the University of Tennessee, for the calculations of the heat flow relationships in the experiment under gamma and electrical heaters anticipated heating conditions; K. R. Thoms of the Reactor Division for making the gamma scan of the ORR E-5 irradiation facility; J. E. Corum of Experimental Engineering for assisting in the design and preparation of working drawings; R. T. Guice and W. T. Coward of the Y-12 Plant Maintenance Department for constructing the capsule; W.K.R. Fennel of the Reactor Division for the maintenance and operation of the experiment; Frances Scarboro of the Metals and Ceramics Division for preparing the manuscript; and the Graphic Arts Department for preparing the drawings.

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1. R. J. Price, *Review of Irradiation-Induced Creep in Graphite Under HTGR Conditions*, GA-A12332 (November 1, 1972).
2. R. J. Price, *Ibid.*
3. W. D. Turner and M. Siman-Tov, *HEATING3 — An IBM 360 Heat Conduction Program*, ORNL-TM-3208 (February 1971).

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