INVESTIGATION OF THE PHYSICAL CHARACTERISTICS OF FLUIDIZED GRAPHITE MODERATORS

by

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ABSTRACT

In a fluidized graphite nuclear reactor concept, a bed of graphite particles, fluidized by an upward flow of helium, could act as the moderator and coolant for a reactor core of solid fuel elements.

This thesis investigated some important physical characteristics of such a fluid bed. The plating of graphite on surfaces within the system was measured using bed temperatures up to 350° C.; no significant plating was observed. The particle size distribution and attrition were determined; the particle size distribution remained constant with increasing temperatures, and the bed attrition was almost nonexistent. The entrainment of fines from the bed was of constant size distribution and was effectively filtered from the helium. Apparatus was assembled and operated to produce a fluid bed and to determine the fluidization parameters of bed height-to-diameter ratio, gas velocity, gas distribution, and bed particle size distribution.

CHAPTER 1

INTRODUCTION

1.1 Description of Fluidization

Fluidization, as an industrial process operation, was practiced in ore processing as early as the sixteenth century, but significant industrial recognition has come only in the last twenty years. Chemical engineering has exploited fluidization processes profitably, and now this phenomenon is developing specific nuclear engineering applications.

To define fluidization, consider a packed bed of finely divided solid material. A fluid moving upward through the bed, at low superficial velocity, flows through the interstices of the bed, with the solid particles remaining fixed in position. As the fluid velocity is increased, the bed volume increases. The frictional drag increases until it becomes high enough to support each particle. At this point, the pressure drop is essentially equal to the weight of the bed. Now that the particles forming the bed are separated by fluid, the bed is unable to resist a shearing force and behaves like a liquid. When a bed of solids exhibits this characteristic, it is termed fluidized.

The fluid bed has an interface, exhibits viscosity, and has film coefficients similar to those of a liquid. It differs, markedly, from a fluid in that the gas fluidized bed density is not greatly affected by temperature between the extreme limits of the gas

condensation point and the solid vaporizing pressure. The particle movement within the bed is generally circular in a vertical plane; the particles rise in the center and descend near the outer edge, with some lateral movement throughout. Some bubbles of gas adhere to the particles and circulate in the same manner.

1.2 Application of Fluid Beds to Nuclear Technology

Fluidization has had important chemical and physical applications in the nuclear fuels and power industry. Also several nuclear reactor or reactor component concepts have suggested applications for fluidization techniques.

Fluid beds of fuel particles may be used as the reactor core, or fluidized solids may be used as moderators, coolants, or both, with solid core elements. Fluidization has also been suggested as an efficient means of controlling reactor power levels, either as a control rod device or by fluid bed density changes.

In a fluidized core, fuel particles are formed as solid balls, and the fluid most frequently used is water. Solid moderator surrounds the upper region of the core, and some power control is exercised by varying the fluid flow and thus bed density.

A modification of the fluidized core concept could involve a mixing of another moderator material with the fuel solids to form a homogeneous core design. Such cores would have control rods of boron or cadmium in the bottom of the vessel as a safety device to absorb neutrons, if the fluid flow rate were suddenly decreased and the fuel settled to the bottom of the vessel.

A different concept, one using a fluid bed as a moderator and

coolant, has many desirable features. Such a system uses graphite particles and helium as the fluid. The core is formed of solid fuel elements placed within a large bed of fluidized graphite. The bed acts as a moderator for the core and is the heat transfer medium from the fuel to steam pipes within the fluidization vessel. The bed is a moderator and consists of only carbon and helium; thus, the neutron economy of the system is excellent. The steam tubes are not in an area of high neutron fluxes; therefore, they may be fabricated from materials used in fossil fuel steam generating systems.

Fluidized reactors, in common with other gas cooled reactors, could result in reduced fuel preparation and fabrication costs. Moreover, their expected excellent neutron economy allows the use of natural or slightly enriched fuel. Fluidized reactor concepts would involve large vessels and systems, but their design is basically simple, with a minimum amount of moving parts and control equipment.

Before a complete analysis of the feasibility of a fluidized moderator nuclear reactor can be made, several factors must be determined that cannot be found in the literature. In the case of graphite and helium, the parameters affecting fluidization must be investigated, particularly in the high temperature ranges. Heat transfer rates, erosion of surfaces, loss of particles by entrainment, changes in particle size and attrition, purification of the helium stream, and plating of graphite on heat transfer surfaces must all be determined for the conditions of a reactor system.

CHAPTER 2

STATEMENT OF THE PROBLEM

This investigation is to determine some essential information about the gas fluidization of graphite, as it might be used in a nuclear reactor. The plating out of graphite on surfaces within the system, particle size distribution, entrainment, and attrition of the bed are to be determined. The particles entrained in the gas will be filtered and observed. This work is to be done over a range of temperatures, limited only by the characteristics of the equipment.

Involved, first, is the design and construction of a containment vessel and gas system suitable to operate a fluid bed and evaluate certain critical parameters for fluidization. These parameters are the height-to-diameter ratio of the fluid bed, the gas velocity, the gas distribution method, and the particle size distribution of the bed. An integral part of this investigation is the testing of the experimental setup, procedure, and measuring apparatus.

CHAPTER 3

FLUIDIZATION TECHNOLOGY

A discussion of the technology of fluidization is appropriate before going into the procedure of these experiments. When the solid particles are completely separated from each other, the fluidization is termed "particulate." Particulate fluidization is normally achieved only when the fluid is a liquid. When a gas is used, the solids are present in the bed, not as individual units, but as aggregates. Thus, gas-type fluidization is termed "aggregative," and no mean free particle path is discernible. The gas rises through the bed as bubbles, with some of the gas clinging to the particles, or aggregates, as they circulate through the bed.[1]

A fluid bed that has a definite upper boundary, with the solids moving freely within it, is in a "dense-phase." If fluid velocities are increased so that solids are entrained and rise upward throughout most of the bed, having no upper boundary, the bed is in the "dispersed suspension" or "dilute fluidized phase." If the particles are entrained in a high velocity gas, one has a "gas-suspension" or "slurry."

"Minimum fluidization" is that point where the fluid velocity is sufficient to expand the bed and allow free movement of particles. There is little, or no, mixing of particles until fluid velocities are

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1. Max Leva, Fluidization, McGraw-Hill Book Co., Inc., 1959, p. 19

increased slightly to further expand the bed and achieve a "turbulent fluidized bed." This bed is still in the dense-phase but has excellent solids mixing and particle circulation.

"Channeling" is a condition within a bed whereby the fluid picks out a preferential path through the solids, leaving the particles out of the stream at rest. This is more prevalent with gas than liquid, and beds made up solely of fine particles are more susceptible to channeling than beds with a distribution of larger particles.

"Slugging" is a condition in which bubbles of the fluid, more frequently gas than liquid, coalesce to a size approaching the diameter of the confinement vessel. The bubbles rise, forming piston-like slugs of solids between bubbles, which also rise to a certain height and then disintegrate. Slugging increases as height-to-diameter ratios and fluid velocities increase. Erosion on surfaces increases, and particle attrition is more noticeable.

Attrition is the gradual reduction of large particles to smaller ones by the grinding action of the bed. Certain amounts of these fines are entrained in the fluid and removed from the bed, even when the bed is in the dense-phase.

Variables affecting fluidization are: particle size distribution, fluid flow rates, the distribution of fluid to the bed, the height-to-diameter ratio, the agitation or vibration of the bed, the shape, size, and density of the solid, the viscosity and density of the fluid, the temperature of the system, and the shape and make-up of the containment vessel. A change in the quality of fluidization will

affect bed density, particle movement, bed turbulence, heat transfer, erosion, attrition, and entrainment.

Considering a nuclear reactor system, changes in bed density, particularly if channeling or slugging occurred in the core area, would be serious. "Quality of fluidization" is a term that describes the degree of homogeneity of the bed.

CHAPTER 4

EXPERIMENTAL PROCEDURE

4.1 Materials

To develop experimental techniques and to define the fluidizing conditions in an appropriate manner, initial experiments used sand and air. The sand was sifted, using Tyler Standard Screens, to determine the size of particles and to separate it according to these sizes. Microscopic examination of the sand showed a rough appearance. All particles which would not pass a 20 mesh sieve were discarded. The following screen sizes were used to classify the sand particle sizes: 20, 42, 60, 80, 100, 115, and 150 mesh. (The appendix contains a table relating Tyler mesh sizes to the opening in inches, millimeters, and microns.) Air to fluidize the sand was supplied by a small air compressor.

When experience had been gained with sand and air, graphite powder and helium were substituted. The graphite was of reactor grade and was donated by the National Carbon Company. It was sifted and separated using the Tyler Screens. The sizes separated were as follows: greater than 42 mesh, less than 42 but greater than 100, less than 100 but greater than 115, less than 115 but greater than 150, and less than 150 mesh. All graphite passed a 20 mesh sieve.

The helium was supplied in large metal bottles. The helium was released through an Airco helium regulator which could regulate

the pressure on the outlet line.

4.2 Equipment

The apparatus consisted of a containment vessel, gas inlet and distribution system, and a gas outlet and filtering system. The bed containment vessel was constructed using a borosilicate tempered glass pipe, 36 inches high, one-quarter inch thick, and with a three inch inside diameter. Aluminum flanges were attached to the ends of the pipe, so that additional plates or flanges could be secured as needed. The pipe was mounted vertically on a wooden frame. The top of the pipe was closed with an aluminum blind flange. Holes were drilled in the flange to provide the gas outlet and equipment access to the bed from the top. Plastic and rubber gaskets were used to seal the vessel at each end. An aluminum blind flange with a centered inlet hole closed the bottom of the vessel.

During most of the experiments, the bottom of the containment vessel was assembled as shown in the picture in Figure 4.1. Normally flat circular distribution plates with small holes drilled in a circular pattern were used to distribute the gas at the bottom of the bed. Tin plates were used in the cold beds and changed to investigate optimum hole size and distribution. For the heated runs, a four-inch-diameter stainless steel distribution plate (shown in Figure 4.1 above the aluminum blind flange gas inlet plate) was drilled and used in the manner determined from previous experiments. The graphite charge was supported by this distribution plate when no gas was flowing. There was an empty space three inches in diameter and one inch high between

FIGURE 4.1

BOTTOM OF THE CONTAINMENT VESSEL AS IT IS ASSEMBLED FOR A HEATED RUN.

The packed bed of graphite contains the temperature measuring probe, the heating coil, and the plating samples. The gas inlet plate is shown resting on the wooden frame. The thermometer in the foreground indicates the helium inlet temperature.

the bottom of the plate and the top of the gas inlet plate.

For several cold runs, funnels were investigated for gas distribution. A metal and a glass funnel were tried, each being secured to the bottom of the vessel by an aluminum flange with a large center hole which fitted over the funnel. Rubber gaskets sealed the lip of the funnel and the glass vessel rim.

A filter was made and attached to the exhaust fitting at the top of the containment vessel by a five foot length of plastic tubing. The filter was constructed from a three-inch-length of four-inch-diameter steel pipe. Two holes were drilled and tapped for one-quarter-inch pipe on the outer rim of the steel pipe. The holes were spaced thirty degrees apart and were fitted with Swagelock fittings, one used as the filter intake and the other to connect a manometer to measure filter pressure. Filter papers were placed across each open face and held in place by masking tape. No tape was allowed to cross the filtering surface of the paper. Wire mesh was placed outside each paper surface to keep the paper from pushing out or rupturing due to pressure. The wire mesh was held firmly by two outer flanges, bolted together across the outer circumference of the steel pipe. The assembled filter may be seen in Figure 4.2 at the extreme left of the picture.

In earlier work, two straight copper tubes were spaced vertically in the bed to measure the pressure drop across the bed. The heating coils were held in place by two quarter-inch-diameter glass tubes with heavy copper wires inserted down the center of each tube. The heating coils were connected across the ends of the copper wires. A stainless

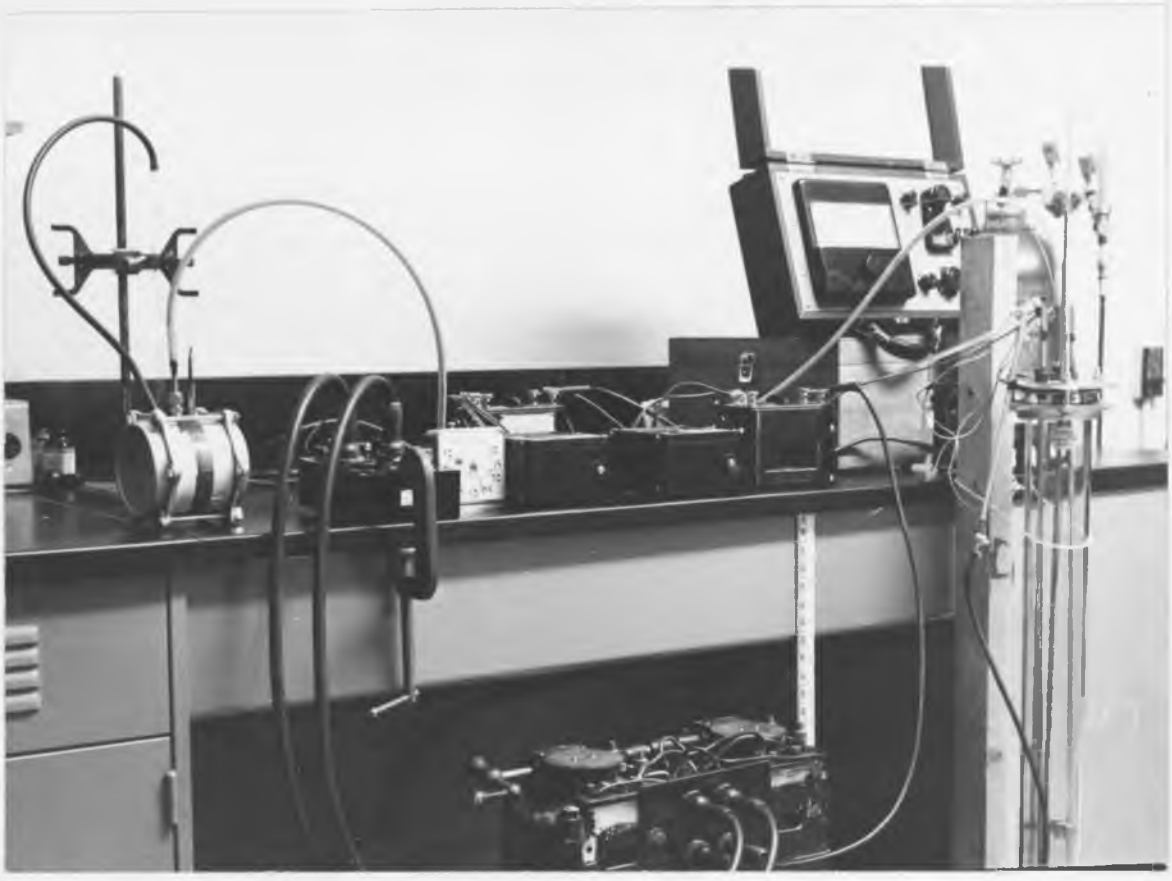

FIGURE 4.2

EQUIPMENT SETUP FOR A HEATED RUN.

Equipment seen on the bench-top from the left: the filter and manometer, junction box, timer, two ammeters, the thermocouple potentiometer, and the helium bottle at the right of bench. In the foreground is the 17 amp variac and the top of containment vessel. Note that the potentiometer is displayed vertically only for the photograph.

steel probe was used to hold samples in place within the vessel.

To measure the amount or effect of graphite plating, the following samples were used:

1. A five inch long stainless steel rod, type 316, of 0.125 inch diameter.
2. 21 and 26 gage steel wires of five inch length with copper electrical connectors attached to each end for assembly.
3. Various nickel-chromium wires of 30, 20, and 18 gage, normally coiled to heat the bed.

The stainless steel rod was held to the sample probe by a set screw, with the two steel wires held bowed between the set screw and the end of the stainless steel rod. The schematic diagram in Figure 4.3 shows the manner of positioning the steel samples within the bed. The samples were arranged at a one-inch-radius from the vessel centerline. The heating coils were of two and three inch coil lengths and could be positioned vertically or horizontally in the center of the vessel at any desired height.

Bed temperatures were obtained with a chromel-alumel type thermocouple attached to a probe that could be raised to any height above the distribution plate and could be rotated from the vessel center to the outer wall. The thermocouple wires were packed in, and insulated by, magnesium oxide powder and were sheathed in stainless steel. The picture in Figure 4.2 shows the equipment setup for a heated run. Rubber and cork stoppers were drilled and fitted over the tubes introduced into the vessel, plugging the holes in the top flange.

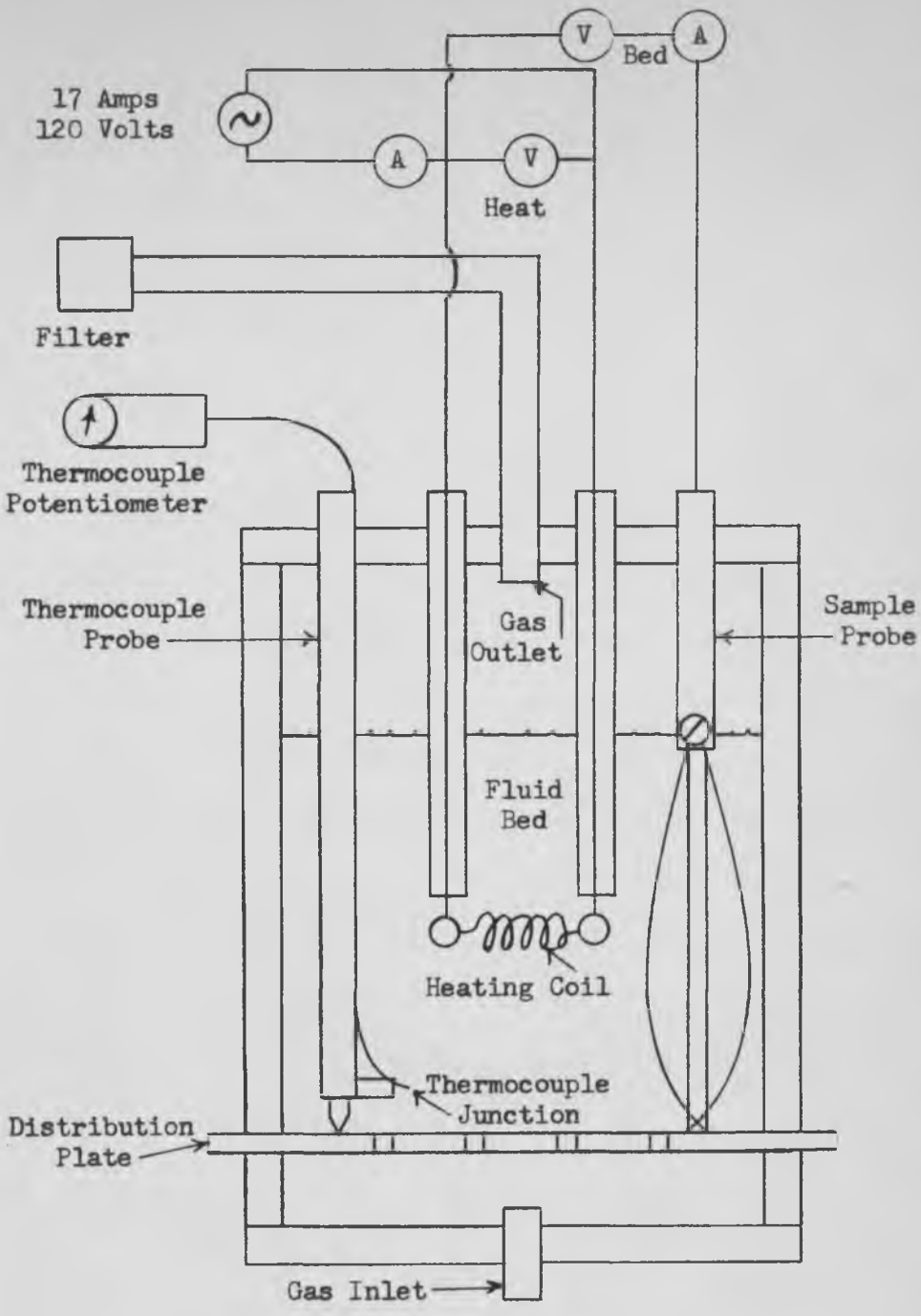

FIGURE 4.3

FLUIDIZATION APPARATUS

Measurement circuits for heated runs are shown. Not to scale.

Rubber-metal cement was used to seal the stoppers into place.

A 17 amp, 120 volt autotransformer was used as the power supply for the heating coil, and two laboratory ammeters and voltmeters were used when necessary. Sample weights were measured on an analytical balance, while heavier weights were weighed on a laboratory balance. Sample resistance was measured on a Wheatstone bridge. Analysis of filter particle sizes was made with a microscope using a screw micrometer eyepiece and a 10X objective lens. Photographs of filter particles were taken with a Universal Camera Microscope "Me F" or metallograph. Helium flow was measured by an orifice-type flowmeter that was calibrated in the laboratory.

4.3 Procedure

Fluid beds were obtained with sand and air to test the equipment and provide data on solids size distribution and gas distribution methods. For a given run, the charge of solids was not altered except by action of the bed. Height-to-diameter ratios were observed for various charges, and the optimum results were noted. Agitation of the bed by mechanical stirrers and pulsing the air supply was attempted, and the effect on fluidization was observed.

The conditions which gave the best results for sand and air were duplicated for graphite beds. Cold runs using air, and later helium, were made to test the graphite particle size composition of the charge, height-to-diameter ratio, and gas distribution system. The entrainment of fines and the attrition of the bed were observed and recorded.

The final phase of this investigation involved heating the graphite and helium fluid bed to observe the quality of fluidization, the plating of graphite, the entrainment of fines, and the attrition of the bed. In preparing for a heated run, all samples were weighed and their resistance was measured before they were mounted on the sample rod and placed in the vessel. All circuits and connections were checked to test measurement circuits and air-tightness. The filter was wiped clean, and two fresh filter papers were weighed and then taped to the filter openings. A graphite charge of known particle size distribution and weight was poured into the vessel after the other apparatus was assembled.

The heated bed runs were made in the following manner. The helium regulator was adjusted to provide 10 psig. on the valve to the flowmeter. The helium flow was varied, until the best fluid bed was observed. The height increase was recorded from packed to fluid bed. The pressure in the filter was recorded from the manometer. Time was started when the bed became fluidized.

Electric current was applied to the heating coil and the current and voltage recorded. Current leakage through the graphite bed to the sample probe was recorded by measuring the current and voltage between one heating coil lead and the sample probe. As the bed reached higher temperatures, the helium flow was reduced to maintain a dense-phase turbulent bed with optimum fluidization quality. The bed temperature was noted on the thermocouple potentiometer, while the thermocouple junction was fixed in position. When the temperature reached a steady state, all instrument readings and the running time

were recorded. Heating current was held constant for each run. After steady state was reached, a temperature profile of the bed was made in accordance with the heat transfer investigation made by Captain Stoneburner. When the desired running time had elapsed, the heating current was slowly reduced to zero with the bed still fluidized. As the bed cooled, the helium flow was decreased and the running time halted.

After each run the samples were removed from the vessel, reweighed, and measured for resistance. The graphite charge was recovered from the vessel and its particle size distribution determined by sifting and weighing. The filter was disassembled and particles trapped inside were scraped onto the filter papers using a spatula. The filter papers containing the graphite were weighed and set aside for later observation with the microscope and metallograph.

The first five heated runs were made with the same graphite charge; the only changes in composition were due to bed action. Then a charge of all fines was used for a heated run; the particles all passed 60 mesh, with over half passing 80 mesh. The final run used the original graphite charge, but the samples were placed above the bed rather than in it. For these last two runs, a one foot length of 18 gage nickel-chromium wire was attached to one end of the vertical heating coil, so that the heating current passed through it in the circuit. This long wire extended above the bed and was heated during these two runs.

CHAPTER 5

DISCUSSION OF RESULTS

5.1 Graphite Plating

When the graphite was fluidized, dust-like particles would coat the surfaces above the bed, though the surfaces within the bed remained relatively clean. These particles had a slippery consistency and adhered to other surfaces and each other readily; however, the apparatus could be wiped clean with a cloth easily. In both cold and heated runs, a fresh charge of graphite fluidized for the first time left an even, heavy layer of these fines above the bed. If the same charge were reused without adding previously unfluidized graphite, there was very little surface dust.

The plating samples turned blue-black in color after one or more heated runs. Any graphite particles that did not shake off the samples due to experimental handling were classified as plating and measured accordingly. For the seven heated runs, and in several other heated fluid bed attempts, a few very large graphite particles, or agglomerations of particles, would cling to the heating coils. Usually these particles or agglomerates were at the ends of the coil between the more closely spaced turns. (An attempt was made to keep the same distance between each turn, though some distortion was caused when mounting the coils on the heating leads or by the action of the bed on measuring apparatus.) These large particles seemed to adhere to the coil as it cooled in the fluid bed. Once steady state temperatures

were reached in the bed, the current and voltage across the heating coil remained constant in all heated runs.

The plating samples were exposed within or above fluid beds of mild turbulence with excellent particle movement. Channeling and slugging were not observed during the heated runs, and the interface resembled the surface of a rapidly boiling liquid.

A resume of the conditions for each heated run is given in Table I. Measurements made to determine graphite plating are consolidated in Table II. The increase in sample weight is an indication of graphite plating on the surface. A consistent change in sample resistance would indicate a surface effect by the graphite or a reaction of the metal with the carbon. The sample diameters were carefully measured with a micrometer, but no significant change could be noted after heating and fluidization exposure.

A graphic picture of the variation in weight of the samples is given in Figure 5.1, along with the variation in weighing obtained measuring procedural control. The fluctuation of sample resistance is shown in Figure 5.2, as well as the variations observed for procedural control.

An experiment was performed to determine the range of procedural control variations due to the following:

1. Laboratory conditions
2. The touching or handling of samples in the experimental procedure
3. Variations in measuring equipment performance and accuracy

TABLE I

CONDITIONS DURING HEATED RUNS

Run Designation:	I	II	III	IV	V	All Fines	VI
Heating Coil Position:	H	H	H	H	V	V	V
Average Bed Temp.(°C):	45	65	60	100	225	240	350
Bed Temp. at Plating Samples, (°C), (measurement distance above distribution plate shown):							
0.5 inches	45						
1.0	45	55	45	120			
1.5	45	65	50	120	240		
2.0	45		45	120	240		
2.5	45		55	95	200		
3.0	45		50	80	185		
3.5			60	95	200		
4.0			70	90	200		
4.5			50	90	205		
Steady State Readings in Amps and Volts:							
Heating Current:	4.2	6.4	6.0	15.0	12.5	12.0	13.0
Heating Voltage:	8.0	18.0	9.0	16.0	26.0	34.0	40.0
Bed Current:	0.15	1.25	1.0	4.0	2.5		
Bed Voltage:	1.0	0.5	0.3	0.7	1.2		
Total Running Time, (min.):	19.1	35.0	26.3	61.5	74.7	45.0	60.0

Notes: 1. Heating coil positions were horizontal or vertical as indicated.
2. The potentiometer battery was weak during Run III.
3. The steel plating samples were placed above bed in final two runs.

TABLE II

CONSOLIDATED GRAPHITE PLATING DATA

Weight of Samples in Grams:

Sample	Original Wt.	Run I	Run II	Run III	Run IV	Run V	All Fines	Run VI
Stainless Steel Rod	7.81229*	7.77966	7.77992	7.77948	7.78110	7.78188	7.78250	7.78748
21 Gage Steel Wire	1.83074	1.83130	1.83100	1.83100	1.83087	1.83178	1.83196	1.83239
26 Gage Steel Wire	1.32126	1.32126	1.32128	1.32145	1.32154	1.32279	1.32274	1.32252
20 Gage NiCr Coil	1.33433			1.33457				
20 Gage NiCr Coil	0.97551				0.98557			
20 Gage NiCr Coil	1.77264					1.77726	1.78078	1.78438
18 Gage NiCr Wire	2.23270						2.23300	2.23300
30 Gage NiCr Coil	0.13636	0.13848						

* Sample included small needle point which broke off before measurement following Run I.

Resistance of Samples in Ohms:

Sample	Original Ω							
30 Gage NiCr Coil	1.7300	1.6870						
Stainless Steel Rod	0.0280	0.0570	0.0470	0.0300	0.0390	0.0490	0.0370	0.0380
21 Gage Steel Wire	0.0560	0.0590	0.0610	0.0550	0.0610	0.0580	0.0550	0.0590
26 Gage Steel Wire	0.1580	0.1630	0.1890	0.1670	0.1600	0.1620	0.1570	0.1570
20 Gage NiCr Coil	1.6260			1.6470				
20 Gage NiCr Coil	1.1950				1.1890			
20 Gage NiCr Coil	2.1220					2.1260	2.1280	2.2030
18 Gage NiCr Wire	0.4810						0.5050	0.5117

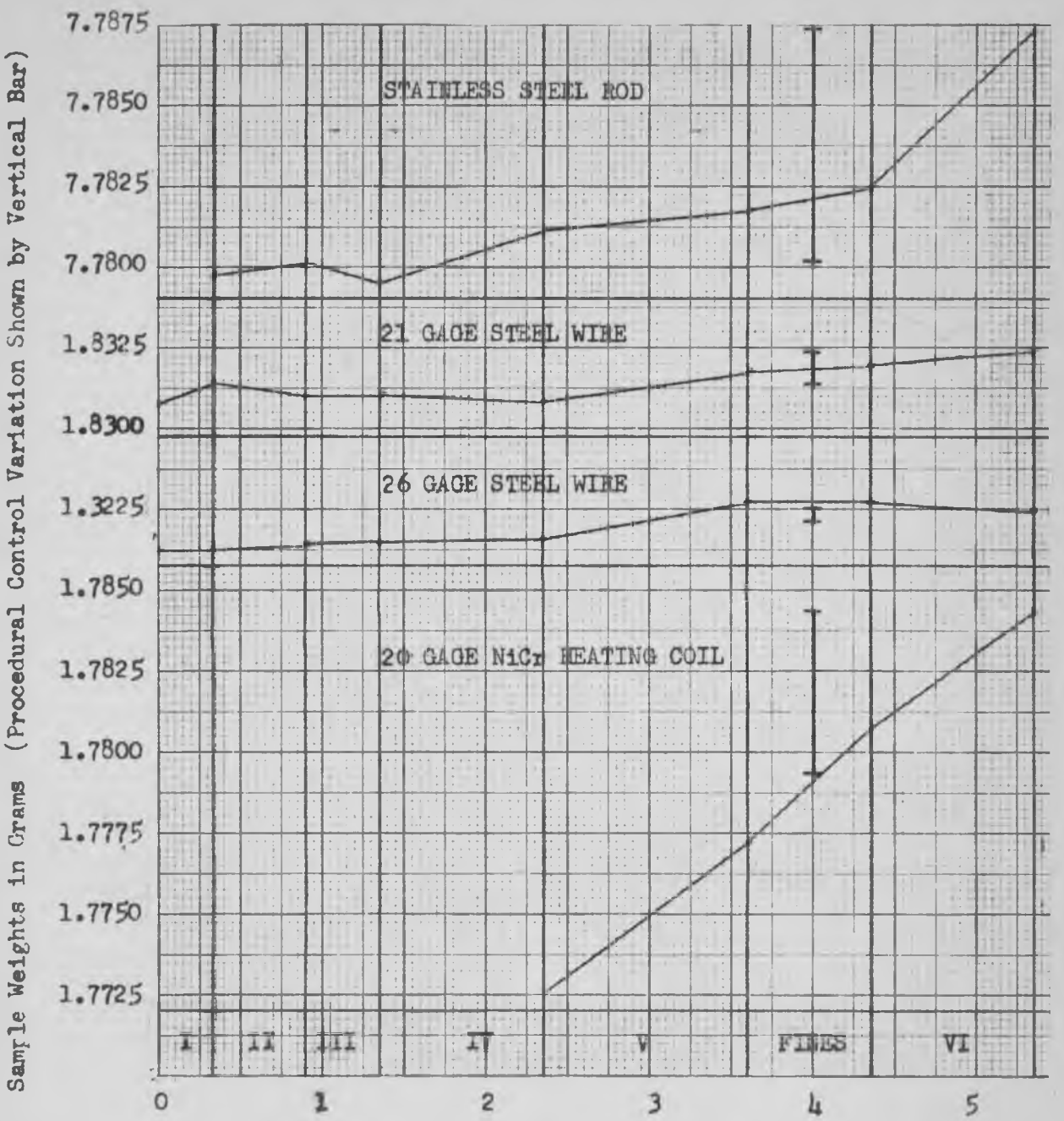

FIGURE 5.1

SAMPLE WEIGHTS AFTER HEATED RUNS

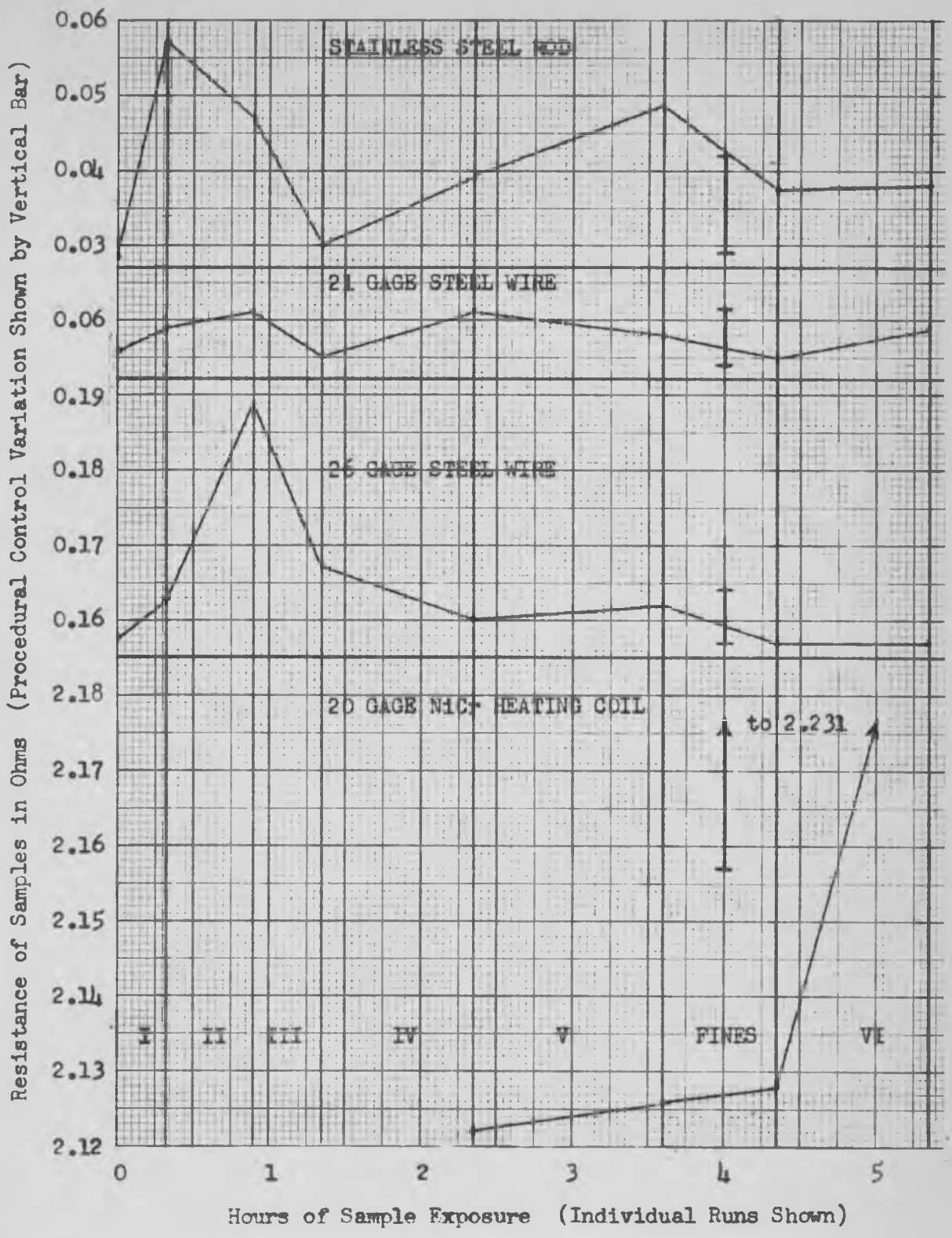

FIGURE 5.2

SAMPLE RESISTANCES AFTER HEATED RUNS

The three primary plating samples and the final 20-gage nickel-chromium heating coil and 18-gage heated sample were allowed to stand in the laboratory for 48 hours following Run VI. Table III shows the results of the weight and resistance measurements, as compared to the data after Run VI. The procedure followed is shown in the table, with maximum observed differences recorded at the bottom of each column.

To provide an analytical calculation of the weight of graphite plating, computations were made assuming the graphite was plated one mil thick on the cylindrical surface of the five inch long stainless steel sample rod. Currie, Hamister, and MacPherson have measured the density of crystalline graphite and obtained a value of 2.26 grams per cubic centimeter. This density was used, assuming the plating is more nearly crystalline than powder, and this value would give a conservative estimate. The graphite would have a volume of 0.001962 cubic inches or 0.03220 cubic centimeters and weigh 0.0728 grams.

During the final three heated runs, the stainless steel rod, the 21-gage steel wire, and the heating coil showed a consistent weight increase. The 26-gage steel wire lost weight slightly during the final two runs. The stainless steel rod gained about 0.0075 grams during the heated runs, approximately 10% of the weight of graphite necessary to achieve a one mil thickness. When the ends of the samples were cleaned in ethyl alcohol, each sample lost a large fraction of the previous weight gain. In the case of the stainless steel rod, this is explained by the fact that the ends were rough, while the side surface was highly polished. For the two steel wires, the terminals

TABLE III

PROCEDURAL CONTROL VARIATIONS

Trial	Sample 1.		Sample 2.		Sample 3.		Sample 4.		Sample 5.	
	Grams	Ohms	Grams	Ohms	Grams	Ohms	Grams	Ohms	Grams	Ohms
1.	7.78748	0.0380	1.83239	0.0590	1.32252	0.1570	1.78438	2.2030	2.23300	0.5117
2.		0.0420		0.0614		0.1633		2.2310		0.5000
3.	7.78205	0.0292	1.83157	0.0580	1.32228	0.1638	1.77975	2.1655	2.23286	0.4983
4.	7.78149	0.0308	1.83139	0.0540	1.32224	0.1603	1.77968	2.1593	2.23294	0.5090
5.		0.0330		0.0566		0.1580		2.1570		0.5020
6.	7.78074	0.0398								
7.	7.78074	0.0345								
8.	7.78012	0.0308								
9.	7.78018	0.0291								
10.		0.0300								
Max. Diff.	0.00736	0.0129	0.00100	0.0074	0.00028	0.0068	0.00470	0.0740	0.00014	0.0134

Trial 1. Results from Run VI.
2. After 48 hour exposure in laboratory.
3. Clean sample terminals in ethyl alcohol and air dry.
4. Handle samples in manner used in equipment setup.
5. Disconnect Wheatstone bridge leads from sample terminals, then reconnect.
6. Clean rod in alcohol, wipe with paper towel, air dry, handle with tweezers.
7. Replace on balance or reconnect leads to terminals and remeasure.
8. Handle sample in manner used in equipment setup.
9. Repeat procedure of Trial 7.
10. Remove and reconnect leads on the Wheatstone bridge.

Sample 1. Stainless steel rod.
2. 21 gage steel wire.
3. 26 gage steel wire.
4. 20 gage NiCr Heating coil.
5. 18 gage, long, NiCr wire.

had much larger surface area than the wire and were made of copper and tin electrical connectors.

A consistent increase in resistance, with increasing weight and exposure, was noted for the heating coil; however, this total increase for the final three heated runs was approximately equal to the resistance variation from room exposure and normal handling deviations. The steel samples did not exhibit any consistent resistance trend compared with weights, and again the variations that did exist in sample resistances were in the range of experimental error deviations.

The blue-black color was completely removed from the stainless steel rod when it was wiped with a paper towel soaked in ethyl alcohol. The weight was decreased to approximately that of the initial sample weight.

In the final two heated runs with the samples above the bed (except for the heating coil), bed action would not help keep them clean, and one would expect an increase in the amount of graphite particles adhering to them. Also the highest temperatures were obtained during these runs. The samples that gained weight during these conditions appeared to do so only on the ends or attached terminals, and the 26-gage steel wire lost weight. Since the current and voltage remained constant during steady state operation of the heating coil, any graphite particle additions must have been made when the coil was cooling.

An F test was made to compare the variance of the plating test weights with the variance of the procedural control weights. Table IV

TABLE IV

STATISTICAL EVALUATION OF PLATING DATA

Samples	Variance of Test	Variance of Control	F	95% Value From F Dist. Table
Stainless steel rod	5.31	9.48	0.531	9.01
21 gage steel wire	2.47	5.84	0.424	234.0
26 gage steel wire	12.28	6.16	2.040	234.0

shows these variances and their ratios which are the F values for this data. The 95% value from the F Distribution Table is shown in the right-hand column of the table. These figures show that there is no significant difference between the plating variances and the control variances.

5.2 Particle Sizes, Attrition, and Entrainment

The distribution of particle sizes in the graphite charge was determined by sifting the charge between runs using a uniform one hundred shakes. The results of the sifting and weighing for the heated runs are shown in Table V. Also shown in the table is the weight of graphite recovered in the filter. The entrainment of particles from the bed was investigated by determining the amount and size of particles trapped in the filter. Bed attrition is observed by determining the loss of large particles and gain in fines, including fines lost by entrainment.

In determining the differences in weights of filter papers, before and after use, the effect of taping the papers to the filter had to be known. In several trials of preparing the filter, but not making runs, an average weight loss of 0.01903 grams was noted, after the masking tape was removed from the filter paper. This loss was added to the weight differences to give the results recorded in Table V.

Initially the effect of fluidization on particle sizes was observed for unheated beds. The graphite charge used was made up of only course and fine particles. Matheson and Hall have noted that a

TABLE V

GRAPHITE PARTICLE SIZE DISTRIBUTION AND BED LOSSES

	Original Charge	Run I	Run II	Run III	Run IV	Run V	Run VI
Average bed temp. in °C:		45	65	60	100	225	350
Time for run in minutes:		19.1	35.0	26.3	61.5	74.7	60.0
Graphite Charge Composition	Grams	Grams	Grams	Grams	Grams	Grams	Grams
Smaller than 42 mesh, Would not pass 60 mesh.)	427.6	406.2	379.8	401.3	403.6	399.7	406.1
Smaller than 60 mesh, Would not pass 80 mesh.)		17.5	38.1	18.3	14.7	16.4	7.9
Smaller than 80 mesh, Would not pass 100 mesh.)		1.4	2.9	1.2	1.7	1.8	1.2
Smaller than 100 mesh.)		0.1	1.0	0.3	0.7	0.7	0.7
Total	427.6	425.2	421.8	421.1	420.5	418.6	415.5
Losses due to handling and entrainment from bed from previous run.		2.4	3.4	0.7	0.6	1.9	3.1
Weight of graphite trapped in the filter.		0.40868	0.12137	0.14910	0.09810	0.29042	0.10042

full distribution from 0 to 500 microns gives poorer fluid beds than one ranging from about 100 to 840 microns. The optimum composition by weight is 25% fines and 75% coarse. The graphite charge closely approximated this condition, including 177.3 grams of particles greater-than-42 mesh and 35.2 grams of less-than-100 but greater-than-115 mesh. This charge was used in several beds of fair-to-good fluidization for a total time of 46 minutes. Air was the fluid and the outlet stream was bubbled through a large beaker of water to allow removal and observation of entrained particles. The following is an analysis, after fluidization, of that graphite charge which was initially just coarse and fine particles:

1. Greater-than-42 mesh = 138.4 grams
2. Passed 42 but not 100 = 33.8 grams
3. Passed 100 but not 115 = 22.6 grams
4. Passed 115 mesh = 6.05 grams

11.7 grams of graphite were lost from the bed by entrainment or in handling.

Another series of cold runs showed a similar change of particle sizes in the graphite charge, and after 90 minutes of fluid bed operations, the fines that had collected in the filter were analyzed. The initial charge was all coarse particles of greater-than-42 mesh; funnels and plates were used for gas distribution. The filter particles had a tendency to agglomerate, so 250 shakes of the screens were used instead of the normal 100. The following weights of particles were obtained.

1. Greater-than-115 mesh = 0.38413 grams
2. Passed 115 but not 150 = 0.46554 grams
3. Passed 150 but not 200 = 1.30250 grams
4. Passed 200 mesh = 1.88280 grams

The result of the analysis of filter particle sizes during the regular heated runs are given in Table VI. The sample was prepared for microscopic and metallograph examination by shaking or scraping graphite from the filter paper onto a glass slide, then tapping the slide until the graphite spread evenly on the surface. The particles would adhere to the surface so that the slide could be inverted without observable spillage. The average particle size shown in Table VI was determined by visually observing the range of particle sizes on the slide, then measuring with the eyepiece the groups visually selected to represent the average size particles.

Figure 5.3 is a photograph made with the metallograph of the filter graphite particlesafter Run VI. Small, average, and large size individual particles are shown, as well as several large masses or agglomerations of smaller particles. These agglomerations were neither counted nor measured in determining the figures shown in Table VI.

The entrainment of particles was especially heavy during the heated run with a charge of all fines. The original charge was made up of half 60 to 80 mesh particles with the other half passing 80 mesh. The total weight of the charge was 388.1 grams. After the 45 minute run, 48.7 grams of graphite were collected in the filter and the whole containment vessel above the bed was black with graphite

TABLE VI

GRAPHITE PARTICLE SIZES IN THE FILTER

	Run I	Run II	Run III	Run IV	Run V	Run VI
Particle Size in Microns						
Smallest particle observed:	0.5	0.7	0.5	0.5	0.8	1.2
Largest particle observed:	80	95	120	80	60	110
Average size range:	8 to 16	10 to 15	11 to 14	12 to 14	11 to 13	12 to 18

FIGURE 5.3

PHOTOGRAPH MADE BY A REICHERT METALLOGRAPH OF THE FILTER GRAPHITE PARTICLES AFTER RUN VI. MAGNIFICATION: 250x

dust.

The graphite entrainment decreases with a decrease in the amount of fines in the bed. For the heated runs there was a decrease in filter graphite weight with the exception of the last two regular runs. The increase here corresponds to notably higher bed temperatures than previously used, though the analysis indicates the particle distribution in the bed and the filter remain essentially constant with earlier heated runs. The heated helium may pick up and entrain more graphite particles above the bed and in the plastic tubing before reaching the filter. A small hill of graphite was observed opposite the inlet part of the filter only after Run V. This extra graphite probably came from the lines since the overall bed loss is low. The heated runs showed very little bed attrition with an almost constant particle size distribution. This general lack of grinding in the heated beds may be explained by several theories. First, there may have been grinding, but an equal amount of agglomeration of smaller particles. Sintering would not take place at these low temperatures and pressures; however, some adhesion of particles is evident both in the filter and the bed. Another possible reason involves the small bubbles of helium that are known to circulate with the solids through the bed. Under heated conditions, these gas bubbles might cling to the particles, acting as cushions to prevent much of the contact between particles. The amount of heat does not seem to be a factor yet, since the particle size distribution remained essentially constant over a range of bed temperatures. Very low helium flow rates were used in the heated beds; yet, the particles have good movement. This makes it appear likely that the gas bubbles, circulating with the

particles, aid the bed movement.

5.3 Fluidization Variables

In general, fair-to-good fluidization could be produced using this equipment setup. The height-to-diameter ratios of 1 to 1.5 were observed to produce more satisfactory beds; it was difficult to achieve a fluid bed if the ratio was below 0.7 and ratios above 2.5 resulted in slugging. The distributions of coarse and fine particles were observed to be better than a distribution of all fines or the use of a charge of uniform sized particles.

Devices were required to give the proper distribution of gas to the bed. Work with distribution plates, funnels, and funnels enclosing plates was done to determine optimum gas distribution. The funnels produced the fastest particle movement within the bed; however, these beds were characterized by a tall spout of graphite spewing up above the bed center. The distribution plates produced more uniform fluid beds without the center spouting. Larger sizes and numbers of holes per plate were investigated, but the most even quality beds were obtained with distribution plates having 0.040 inch diameter holes spaced slightly more than one per square inch of plate surface. This design was used on the distribution plate for the heated runs.

Fluid flow rates were low; the air velocities ranged from 1 to 2 feet per second while the helium flows during the heated runs ranged from 0.13 to 0.80 feet per second. Above minimum fluidization, the flow rates could be varied as much as 0.5 feet per second without visibly affecting fluidization quality. The lowest flow rates producing

acceptable fluid beds were used for all runs.

Physical agitation of the bed increased the quality of fluidization and allowed minimum fluidization to occur with lower fluid velocities. Agitation was accomplished by pulsing the air flow from the compressor or by stirring the bed while in operation.

A graphite density of 2.065 grams per cubic centimeter was measured by displacement of methyl alcohol.

CHAPTER 6

CONCLUSIONS

Significant graphite plating was not observed on the surfaces of the steel samples at bed temperatures up to 350°C. The blue-black film on the steel was due to the presence of carbon, but was not effectively observed except on the stainless steel sample. The weight of the film is not sufficient to be a reliable indication of graphite plating. Several large graphite particles or masses did adhere to the nickel-chromium heating coils causing a resistance increase. These particles did not become attached while the coil was operating at steady state temperatures. The maximum increases in weight due to graphite plating were 0.00782 grams for the stainless steel, (corresponding to less than 0.0001 inch thickness), and 0.0117$\frac{1}{4}$ grams for the heating coil. Further investigations of plating must be made at higher temperatures.

For heated beds of graphite and helium, attrition is very slight with the particle size distribution within the bed and entrained in the helium being constant. This equipment can produce good fluid beds and be used to determine certain critical fluidization parameters.

Because of bed conduction and the leakage of heating current between the heating coil terminals, a bed current and voltage were observed between the sample rod and a heating lead. It is not known to

what extent this current affected graphite plating on the samples. A heating device that is insulated from the bed would eliminate current leakage to the bed.

A systematic timetable was not followed after each run so that there was some variation in the time a sample remained in a graphite packed bed before measurements were made. This exposure variation could have resulted in a procedural error in sample weight or resistance. A trial procedure is necessary after, as well as during, future experiments of this nature. Included in the procedure should be a uniform cleaning of sample ends and terminals before measurements.

Variations in particle size measurement may be regulated by increasing the number of sieve shakes per screening. The percentage of smaller particles that do not fall through a larger screen opening will decrease with each additional shake up to a limiting value.

Little is mentioned in the literature concerning fluidizing graphite with helium, so most of the known data on graphite plating comes from gas-suspension work, whereby very small particles - average size of 5 microns or less - are transported with high gas velocities and pressures. In their investigation of gas-suspension coolants for power reactors, Walsh, et.al., reported the plating of graphite on heat transfer surfaces. Moreover, the graphite literally piled up, or plated, on some surfaces within pumps, valves, and lines. The plating differences noted between this investigation and the suspension investigation may be explained by considering 1) the reduced gas velocity and pressure; 2) the increased particle sizes; and 3) the lower temperatures compared to the gas-suspension data mentioned above.

The lack of attrition in the heated beds tend to confirm the reports concerning the circulation of bubbles of fluid with solid particles in fluid beds.

The optimum fluidization parameters observed in this investigation conform in general to the results described by Leva or Zenz and Othmer. For the distribution plate design, a slightly smaller hole size and 25% more of them gave better results than similar designs reported by Leva. The ratio of coarse to fine particles did not appear as critical for good fluidization as given in Zenz and Othmer's book; however, the charge of fines gave poor results as predicted.

CHAPTER 7

FUTURE WORK

The experiment program should continue until heating sources for the bed approach core temperatures appropriate for a fluidized graphite nuclear reactor. Graphite plating, particle size, attrition, and entrainment must be determined.

The parameters of fluidization observed in these experiments may be extended to future work, and improvements in the fluid bed should be attempted. Baffles, bed agitation, and vibration should be tried to determine the effect on fluidization quality and on heat transfer characteristics of the bed. In general, anything that improves fluidization should improve the bed heat transfer data, but there is a special reason for investigating the effect of baffles on the heat-producing surfaces in the bed. Some density fluctuation is normal in gas fluidized beds, and this could lead to hot spots along a heat element, especially if channeling were to occur. Baffles or other bed agitation might control, or limit, density changes and channeling.

The temperature limits of the glass containment vessel have been reached, and an iron vessel of four-inch inside diameter is available to build apparatus for higher temperature work. With the higher temperature, there will be a requirement for vessel insulation to increase bed temperatures and to hasten steady state conditions.

Equipment improvements and additions should be made, including

the use of more thermocouples, a pump to recirculate the helium, and more samples and heating sources. These would allow greatly increased operating times, improve the quantity and quality of data obtained, and facilitate an investigation of the filtering, purification, make-up, and pumping requirements for a complete gas system.

A further extension of this work would be the design and construction of a higher velocity and pressure system. Such a device might bridge the gap between fluid beds and gas suspensions, and it could allow possible correlations of data from either end of the gas-solids spectrum.

APPENDIX

OPENING SIZES FOR TYLER STANDARD SCREEN SCALE

MESH	OPENING IN:		
	Inches	Millimeters	Microns
20	0.0328	0.834	834
42	0.0138	0.351	351
60	0.0097	0.246	246
80	0.0069	0.175	175
100	0.0058	0.147	147
115	0.0049	0.124	124
150	0.0041	0.104	104
200	0.0029	0.074	74

SELECTED BIBLIOGRAPHY

1. Astley, E. R. "Heterogeneous Lattice Fluidized Bed Reactor Study," General Electric: HW-48847, March 5, 1957.

2. Benenati, R. "Pebble-Bed Reactor," Symposium on Particle-Fluid Interactions, TID-7592, May, 1959.

3. Currie, L. M., Hamister, V. C. and MacPherson, H. G. The Production and Properties of Graphite for Reactors. The National Carbon Company, 1955.

4. Dixon, Wilfrid J. and Massey, Frank J., Jr. Introduction to Statistical Analysis. New York, McGraw-Hill Book Company, Inc., 1951.

5. Friedlander, S. K. "Behavior of a Dilute Turbulent Suspension," Industrial and Engineering Chemistry, 49: 1151-6, 1957.

6. Hoel, Paul G. Introduction to Mathematical Statistics. 2d edition. New York, John Wiley and Sons, Inc., 1954.

7. Kelly, V. P. "Hydraulic Studies for the Fluid-Bed Reactor," Nuclear Science and Engineering, 10: 40-4, 1961.

8. Leva, Max. Fluidization. New York, McGraw-Hill Book Company, Inc., 1959.

9. Luckow, W. K., Thomas, G. R. and Helms, T. C. "Gas-Suspension Coolant Development Project," Babcock and Wilcox Company, Atomic Energy Division: BAW-1186, Feb., 1960.

10. Matheson, G. L. and Hall, H. J. United States Patent 2,711,387, June, 1955.

11. McAdams, William H. Heat Transmission. 3d edition. New York, McGraw-Hill Book Company, Inc., 1954.

12. Miller, D. "Fluidized-Bed Studies at Argonne," Symposium on Particle-Fluid Interactions, TID-7592, May, 1959.

13. Post, Roy G. Fluidized Moderator Reactor Concept. An unpublished paper by Dr. Post, Department of Nuclear Engineering, University of Arizona, Tucson, Aug. 2, 1961.

14. Post, Roy G. "Fluidized Reactors," Symposium on Current Trends in Nuclear Power. Tucson, University of Arizona and Argonne National Laboratory, Feb., 1962.

15. Post, Roy G. "Report of Invention, Fluidized Moderator Reactor," General Electric: HW-49506 (HW IR-851), April 2, 1957.

16. Reed, T. M., III and Fenske, M. R. "Effects of Agitation on Gas Fluidization of Solids," Industrial and Engineering Chemistry, 47: 275-82, Feb., 1955.

17. Rowe, P. N. and Wace, P. F. "Gas-Flow Patterns in Fluidized Beds," Nature, 188: 737-8, 1960.

18. Scheve, M. "Liquid Fluidized-Bed Reactor," Symposium on Particle-Fluid Interactions, TID-7592, May, 1959.

19. Schluderberg, D. C. "Gas-Suspension Reactor Coolants," Babcock and Wilcox Company, Atomic Energy Division: BAW-1182, Dec., 1959.

20. Stoneburner, John F. Investigation of Fluidized Reactor Systems. Master of Science Thesis, University of Arizona, Tucson, 1962.

21. Walsh, E. E., Rhode, G. K., Roberts, D. M. and Schluderberg, D. C. "Gas-Suspension Coolants for Power Reactors," Symposium on Particle-Fluid Interactions, TID-7592, May, 1959.

22. Zenz, F. A. and Othmer, D. F. Fluidization and Fluid-Particle Systems. New York, Reinhold Publishing Corp., 1960.