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AN INVESTIGATION OF SOME OF THE CAUSES
FOR THE SPALLING OF IMPREGNATED GRAPHITE

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Introduction

One of the important problems with respect to the low permeability impregnated graphite is its tendency to spall during manufacture. In order to prevent spalling of the graphite, it is necessary to determine, at least to some degree, the causes of spalling. One of the ways to enhance the knowledge on the causes of spalling is to determine the behavior of the impregnant - graphite system during the entire process employed in making low permeability graphite from more porous graphite. As a consequence, a limited experimental program was undertaken in the following general areas:

- (1) An analysis of the variables in the graphite base stock and in the manufacturing process of impregnated graphite.
- (2) Metallographic examination of impregnated graphite heated to various temperatures and at various stages in the process.
- (3) Gas evolution characteristics of impregnated graphite under conditions similar to those employed for obtaining low permeability graphite from impregnated graphite.

Graphite Base Stock and the Manufacturing Process of Impregnated Graphite.

In general, spalling is due either to 1) the properties of the graphite base stock, or to 2) one or more of the process steps used in making the impregnated graphite. Before any experimental work described in this report was undertaken a thorough review and analysis was made of the performance of the various graphite base stocks supplied by the vendors and the manufacturing of impregnated graphite at General Atomic.

For purposes of providing a background, the impregnation process of the graphite used in this investigation will be described briefly. For details of the impregnation process reference is made to a report entitled, "A Process for Making Graphite Impermeable to Gases at High Temperatures" by G. R. Tully, Jr. and B. F. Disselhorst, (GA-2318).

The impregnation process consisted of the following basic steps: impregnation, "blow through", "cure", carbonization and graphitization. A brief description of each process step is given below.

(1) Impregnation - A graphite body (typically a tube with a 3/8 inch wall thickness, 3-1/2 inch outside diameter and 4 feet in length) was placed in a steel chamber and evacuated. A liquid monomer was then injected into the system until the graphite was totally immersed in the monomer. The monomer was a mixture of 10 mols of furfuryl alcohol and 1 mol of maleic anhydride or of the same mixture containing an amount of ethyl cellulose varying from 5% to 15% by weight. The monomer was impregnated into the graphite by a series of pressure - (1/2 hour at 150 psi) vacuum (1/2 hour at minus 30 inches Hg.) cycles.

(2) "Blow through" - The graphite tube containing the liquid monomer was subjected to a pressure difference of approximately 50 psi between the inner and outer walls of the tube in order to force out some of the monomer trapped in the interconnected passages.

(3) "Cure" - The monomer in the graphite was "cured" or polymerized by maintaining the impregnated graphite in a typical cure cycle at 60°C for 16 hours and at 100°C for 5 hours. Pressures as high as 80 psi were sometimes required when the impregnated graphite was "cured" at 60°C in order to prevent the monomer from exuding from the graphite.

(4) Carbonization - The degradation of the polymer was achieved by means of a heating cycle. The length of a typical heating cycle was two days and consisted of a heating rate of 13°C/hr from 100°C to 500°C followed by a heating rate of 50°C/hr from 500°C to 900°C. Carbon and gases, such as water, CO, CO₂, H₂, N₂ and hydrocarbons were the products of degradation. The gases formed were intermittently released from the chamber whenever the pressure was greater than 10 psi. It should be pointed out that some of the gases formed were either adsorbed in or trapped in the graphite, consequently, additional outgassing took place when the graphite was heated to higher temperatures.

(5) Graphitization - Graphitization was achieved by heating the carbonized body rapidly (~ 600°C/hr) in a helium atmosphere) to the temperature range of 2600°C - 2800°C and maintaining the graphite in this temperature range for a minimum of 30 minutes.

A summary of the spalls which occurred in the various graphites which were impregnated appear in Table I. For greater detail about the history

TABLE I

Summary of Spall Occurrences

Type of Graphite	Source	No. of Graphite Specimens	No. of Carbon-ization Cycles	Spalls			Cause Unknown	% of carbonization cycles resulting in spalling
				Total No.	No. due to process	No. due to graphite		
901-S	Speer	33	105	7	6	-	1	7
HLM-85	Great Lakes	14	45	3	3	-	-	7
NCC	National Carbon	7	11	6	2	4	-	55
HC-26	Great Lakes	12	27	5	-	5		23
EYX-60 and EY-9	Morgan	5	5	2	2	-	-	40
OS	Great Lakes	4	10	1	1	-	-	10
Total		75	203	24	14	9	1	12

and results on spalling at General Atomic, the reader is referred to a GA memorandum from G. R. Tully to R. A. Meyer, "Spalls in Impregnated Graphite", December 27, 1960.

The first three out of a total of seven cases of spalling of 901-S impregnated graphite established the length of the carbonization cycle during the first two impregnation and the need for a "blow through". Three of the four remaining spalls were attributed to a heating rate which was too rapid during the later carbonization cycles. In the seventh case, spalling occurred in spite of a slower heating rate during the fourth carbonization cycle. The cause of the latter spall was not apparent.

Although three cases of spalls occurred in HLM-85 impregnated graphite, only one of the cases proved significant. It further established the need for a "blow through". The remaining two cases were due to a malfunctioning of the equipment.

Six cases of spalls were observed with NCC special grade base stocks which were impregnated. Two occurred during the first carbonizing cycle and resulted from the lack of a "blow through". The remaining spalls occurred during the second carbonizing cycle and were due to the laminar weakness in the graphite base stock and or to an overloading of the graphite base stock with impregnant.

In every instance where impregnated graphite base stock HC-26 was used, a failure resulted. The cracks in the graphite indicated die spider weakness in addition to pin holes. These failures were clearly due to the graphite base stock.

One case of spalling in EYX-60 impregnated graphite was due to the heavier than usual wall of the graphite specimen which was difficult to outgas and the second was attributed to a rapid heating rate during the carbonization cycle.

The limited work on OS graphite resulted in a spall during the third carbonization cycle. The spall was due to a lack of a "blow through" in the earlier cycles.

METALLOGRAPHIC EXAMINATIONS OF IMPREGNATED GRAPHITE

Apparatus and experimental procedure - Polished graphite specimens were examined at room temperature using bright field metallographic techniques with a Bausch and Lomb Research Metallograph. The general areas of investigation of graphites included examinations of:

- 1) graphite base stocks
- 2) impregnated graphite after curing only
- 3) impregnated graphite after curing and carbonizing.
- 4) impregnated graphite after graphitizing.
- 5) graphite impregnated with a mixture of furfuryl alcohol and maleic anhydride and graphite impregnated with the furfuryl alcohol - maleic anhydride mixture containing various quantities of ethyl cellulose.
- 6) regions of impregnated graphite which had spalled during carbonization or graphitization.

Results - Photomicrographs (300x) of a typical set of the results obtained appear in figures 1a, 1b, 1c and 1d. The captions below each photomicrograph briefly describe the treatment received by the graphite specimen. A discussion of these results is undertaken later in this report.

In addition to the examinations performed employing the usual bright field metallographic techniques, polarized light and the sensitive tint techniques were also used. However, the latter techniques did not yield any clearer definition of the graphite than the bright field technique.

GAS EVOLUTION CHARACTERISTICS OF IMPREGNATED GRAPHITE

Apparatus and experimental procedure - An outline of the apparatus and experimental procedure of the gas evolution characteristics of impregnated graphite will be given here and reference is made to the appendix for further details.

HLM-85 graphite was selected for these studies because results reported earlier showed that this graphite was adaptable for impregnation. Specimens were cut from one common piece of HLM-85 graphite base stock which was impregnated up to and including the "blow through" and "cure" steps by a process similar to that described earlier. The specimens were weighed and

Base stock (300x) - Permeability: $1 \times 10^{-1} \text{ cm}^2/\text{sec}$

After first impregnation and carbonization (300x) Permeability: $2 \times 10^{-2} \text{ cm}^2/\text{sec}$

Figure 1a - Photomicrographs of a typical graphite base stock before and after the first impregnation.

After second impregnation and carbonization (300x) Permeability: $3 \times 10^{-3} \text{ cm}^2/\text{sec}$

After third impregnation and carbonization (300x) Permeability: $4 \times 10^{-4} \text{ cm}^2/\text{sec}$

Figure 1b - Photomicrographs of a typical graphite after the second and third impregnations and carbonizations.

After graphitization following third carbonization (300x) Permeability: $2 \times 10^{-3} \text{ cm}^2/\text{sec}$

After fourth impregnation and carbonization (300x) Permeability: $1 \times 10^{-4} \text{ cm}^2/\text{sec}$

Figure 1c - Photomicrographs of a typical graphite after a third carbonization and a fourth impregnation and carbonization.

After final impregnation and carbonization (300x) Permeability: $2 \times 10^{-5} \text{ cm}^2/\text{sec}$

After final graphitization (300x) Permeability: $4 \times 10^{-5} \text{ cm}^2/\text{sec}$

Figure 1d - Photomicrographs of a typical graphite after a final impregnation, carbonization and graphitization.

an impregnated graphite specimen was inserted into each of nine Inconel capsules which were connected to a common pyrex glass tube. The capsules were evacuated to a pressure of 10^{-4} mm. prior to the initiation of the heating cycle. The heating cycle used was similar to the cycle used normally for carbonizing the monomer in a first cycle impregnation to treat graphite in order to lower its permeability and is given in the following table.

TABLE II

Heating cycle for impregnated graphite specimens

<u>Temperature Range °C</u>	<u>Heating Rate °C/hr</u>
25 to 100°	40
100 to 500°	13
500 to 900°	50

During the heating cycle one of the capsules was withdrawn from the furnace at every 100°C temperature interval in the 100° - 900°C temperature. Immediately prior to the removal of the capsules from the furnace a gaseous specimen was withdrawn.

After the heating cycle was completed, the metal capsules were cut open, the graphite specimens were weighed and the permeabilities of the graphite specimens were measured. (The permeability of a graphite specimen which was not subjected to the heating cycle was also measured.)

Table III lists the weights of the graphite specimens prior to the heating cycle and the percent changes in weight of each specimen after heating it to a particular temperature. Figure 2 is a plot of percent change in weight versus the temperature to which the graphite was heated.

Figure 2 - Temperature °C at which capsules were withdrawn

TABLE III

Wt of graphite prior to heating (gms.)	Temperature °C to which graphite was heated	Percent Weight change in graphite
4.56330	100	0.11
4.26932	200	0.15
4.22956	300	1.29
4.38948	380	2.23
4.61249	500	3.07
4.26581	600	3.49
4.31928	700	3.59
4.64598	800	3.21
4.23530	900	3.62

Table IV contains the permeabilities of the graphite specimens and the respective temperatures to which the specimens were heated. Figure 3 is a plot of these data.

TABLE IV

Temperature °C to which graphite was heated	Permeability (cm ² /sec) at 25°C
25	1.2×10^{-4}
100	1.6×10^{-4} ; 2.1×10^{-4} *
200	1.0×10^{-5} ; 3.0×10^{-5} *
300	2.4×10^{-3} ; 2.9×10^{-3} *
380	1.4×10^{-3}
500	4.2×10^{-3}
600	7.0×10^{-3}
700	1.3×10^{-2}
800	2.0×10^{-2}
900	8.2×10^{-2}

* The permeabilities of these graphite specimens were redetermined to establish the reproducibility of the data due to the apparent large decrease in permeability of the specimen heated to 200°C.

The gaseous samples collected at the various temperatures were analyzed by means of a mass spectrometer. In most instances two collecting flasks were used to obtain the gaseous samples at the respective temperatures thus enabling one of the flasks to be used for the analysis of water only and the other flask for the analysis of the remaining components. When the gaseous specimen was collected in one flask, an analysis of all the components except water was performed.

The results of the gaseous analyses and the temperatures at which the samples were withdrawn are given in Table V.

Table VI is simply a revision of Table V. It should be noted that the values for the various gaseous species at 400°C given in Table V represent the composite total of the gases present in the capsules at which gaseous samples were subsequently withdrawn from the remaining capsules at 500° , 600° , 700° , 800° , and 900°C . Assuming similar gaseous composition in the various capsules, it then follows that the values at 400°C can be divided by five to yield the gaseous composition in each capsule. Such values are given in Table VI at 400°C . Furthermore, assuming that no reaction takes place between the various gaseous species and or the graphite, the values listed in Table V at 400°C were added to all the values obtained at subsequent temperatures.

In an attempt to gain further insight into the changes which take place in the impregnated graphite during the carbonization cycle, portions of the graphite specimens used for studying the gas evolution characteristics were examined employing metallographic techniques. Photomicrographs (400x) of the graphite specimens with appropriate captions appear in figures 4a, 4b, and 4c.

DISCUSSION OF RESULTS

Manufacturing Process of Impregnated Graphite - Early evidence (a) based on a preliminary analysis of the process variables indicated that the amount of impregnant introduced (impregnant loading) during the fourth impregnation cycle could be a critical factor in causing spalls in the manufacturing process. Subsequent results on 30 graphite specimens showed that impregnant loading during the fourth impregnation cycle was indeed a

(a) Progress Report, G. R. Tully to J. F. Schumar, Dec. 28, 1960.
"Supplement to Progress Report for Dec., 1960.

TABLE V

Gas Evolution Products of Impregnated Graphite

Temperature °C at which gaseous products were withdrawn	H ₂ μ moles	CH ₄ μ moles	CO and or N ₂ μ moles	O ₂ μ moles	CO ₂ μ moles	Hydrocarbons μ moles	H ₂ O μ moles
100	0.00256	0.00356	0.0646	0.01570	0.0540	0.0028	214
200	0.0484	0.0212	0.392	0.01492	0.452	0.022	790
300	0.0594	0.0782	0.934	0.1492	2.74	0.062	1234
380	3.08	6.98	19.68	0.0216	37.0	1.28	3500
400 ⁽¹⁾	4.41	45.3	166.0	0.091	281.0	10.0	n.d.
500	6.42	52.2	39.0	0.026	17.96	1.38	256
600	10.72	85.2	25.4	0.026	42.0	0.24	176
700	28.4	53.0	14.32	0.0874	14.84	trace	144
800	73.4	34.8	49.8	0.0456	14.86	0.007	66
900	108.4	54.4	63.4	0.022	18.66	0.30	n.d.
900 ⁽²⁾	13.68	0.1004	2.92	0.0078	1.286	0.02	n.d.

n.d. - not determined

(1) - At 400°C, gases were withdrawn from the remaining five capsules into a common container. These capsules were then subsequently heated to higher temperatures.

(2) - Gaseous products withdrawn after the graphite containing capsule was maintained at 900°C for 1 hour.

TABLE VI

Revised Values for Table V

Temperature °C at which gaseous products were withdrawn	H ₂ μ moles	CH ₄ μ moles	CO and or N ₂ μ moles	O ₂ μ moles	CO ₂ μ moles	Hydrocarbons μ moles	H ₂ O μ moles
100	0.00256	0.00356	0.0646	0.01570	0.0540	0.0028	214
200	0.0484	0.0212	0.392	0.01492	0.452	0.022	790
300	0.0594	0.0782	0.934	0.1492	2.74	0.062	1234
380	3.08	6.98	19.68	0.0216	37.0	1.28	3500
400 ⁽¹⁾	0.88	9.06	33.2	0.018	56.2	2.00	n.d.
500	7.30	61.26	72.2	0.117	74.16	3.38	256 ⁽²⁾
600	11.60	94.20	58.6	0.0440	98.2	2.24	176 ⁽²⁾
700	29.82	62.06	47.52	0.105	71.04	2.00	144 ⁽²⁾
800	74.28	43.86	83.0	0.063	71.06	2.007	66 ⁽²⁾
900	109.28	63.46	96.6	0.040	74.86	2.30	n.d.

n.d. - not determined

(1) - Values in Table V at 400°C divided by 5. These values were added to each of the values at 500°, 600°, 700°, 800°, and 900°C given in Table V.

(2) - Values given are the same as those in Table V, since the amount of H₂O was not determined in the gaseous specimen withdrawn at 400°C.

Impregnated graphite before heat treatment (400x)

Impregnated graphite after heating to 100°C . (400x)

Impregnated graphite after heating to 200°C. (400x)

Impregnated graphite after heating to 300°C. (400x)

Figure 4a - Photomicrographs (400x) of impregnated graphite in the first stages of heat treatment.

Impregnated graphite after heating
to 380°C (400x)

Impregnated graphite after heating
to 500°C (400x)

Impregnated graphite after heating
to 600°C (400x)

Impregnated graphite after heating
to 700°C (400x)

Figure 4b - Photomicrographs (400x) of impregnated graphite at the intermediate stages of heat treatment.

Impregnated graphite after heating to 800°C. (400x)

Impregnated graphite after heating to 900°C. (400x)

Figure 4c - Photomicrographs (400x) of impregnated graphite in the final stages of heat treatment.

critical factor. For impregnant loadings of 2.8% by weight and above, 7 out of 10 graphite specimens suffered spalling regardless of graphite geometry, monomer viscosity, carbonization rate, or initial permeability. For impregnant loadings of 2.6% by weight and lower, no spalls were found in 20 graphite specimens.

Metallographic Examinations - A bright field metallographic technique with a lower limit of definitive resolution of 3μ was used to determine the interaction between the impregnant and the graphite base stock. The results showed that:

- 1) The impregnant enters and occupies the pores of graphite having diameters between 3μ and 40μ during the first and second impregnations (see figures 1a and 1b). No appreciable amount of the volume of the larger pores was found to be occupied by the impregnant until the third and fourth impregnation (see figures 1b and 1c).
- 2) The impregnant did not adhere well to the graphite base stock and appeared in the form of "islands" or "peninsulas" with respect to the graphite base stock (see figures 1b and 1c). Since the metallographic technique provides for viewing only a plane, it was not possible to determine the extent of bonding between the impregnant and the graphite base stock at points other than the plane of viewing.
- 3) During the carbonization cycle, i.e. the 100° - 900°C temperature range, the most significant changes in the impregnant occur in the 100° - 600°C temperature range (see figures 4a and 4b). Virtually all of the carbon that is formed as a result of the degradation of the impregnant takes place in this temperature range. However, a slight densification of the impregnant occurs in the temperature range of 600° - 900°C . (see figures 4b and 4c).
- 4) No change was apparent in the impregnant after the carbonized impregnated graphite was heated to graphitization temperatures (2600° - 2800°C). Furthermore, x-ray diffractions patterns of the impregnant heated to graphitization temperatures in the absence of a graphite matrix revealed that the carbon resulting from the impregnant was only slightly graphitized. ^(b)

(b) Result reported by G. Engle - General Atomic, April, 1961.

5) Considerable non-uniformity was present in the various graphite base stocks with variation in the size and distribution of pores existing in various portions of each piece of graphite as well as in separate pieces of the same grade of base stock graphite. However, the observations did not reveal any correlation between the non uniform pore structure of the graphite base stock and the occurrence of spalls.

Permeability Measurements - The permeability of the impregnated graphite was found to increase with the temperature to which the impregnated graphite had been heated (see figure 3). A notable exception was the graphite specimen heated to 200 °C where the permeability decreased significantly. It is likely that in the temperature range 100° - 200°C the impregnant undergoes further curing which results in a more effective blocking of the pores than that which takes place in the 25° - 100°C or the 200° - 300°C temperature ranges. It is not clearly evident, however, from the photomicrographs (see figure 4a) that more effective blocking of the pores has taken place in the 100° - 200°C range but it is clear from the photomicrographs (see figures 4b and 4c) that less effective blocking of the pores taken place in the 400° - 900°C temperature range resulting in an increased permeability.

Gas Evolution - The results in table V and VI are semi-quantitative and complex in nature and consequently, do not lend themselves towards a postulation of a reaction mechanism. However, the results reveal that the most significant changes take place in the temperature range of 100° - 600°C, with the major portion of the gases evolved in the temperature range of 300° - 600°C. Most of the water, the most prevalent product resulting from the degradation of the impregnant, is formed between 100° and 400°C. However, water continues to be a product at least up to 900°C. The results also indicate that it is likely that reactions take place between the gaseous products formed above 700°C.

Hydrogen was evolved in increasing amount with increasing temperatures, however, most of the gases resulting from the degradation of the impregnant are formed at 900°C or below (see table V).

Weight Changes - Figure 2 shows that the most significant weight changes occur in the 100° - 600°C temperature region, with the major portion of the change taking place in the 300° - 600°C. temperature range. These results are in qualitative agreement with the gas evolution results as well as those changes observed using metallographic techniques.

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Conclusions

Several conclusions can be drawn from the investigations performed.

- 1). Graphite base stock - Greater standardization and uniformity in pore size distribution than presently available is desirable. Such a condition would result in a firmer control over the impregnation process.
- 2). Impregnant - The type of impregnant employed is important. The present investigation was not only limited as to the type of impregnant but in addition the knowledge of the mechanism of polymerization of the impregnant is meagre. A more extensive study should be undertaken of the behavior of various impregnants as a function of process variables. Also, other impregnants and or additives to the impregnant which may result in better adherence of the impregnant to the walls of the graphite and which might aid graphitization deserve attention.
- 3) It is clear from the photomicrographs, the weight and permeability changes and the gas evolution characteristics of the impregnated graphite that the most significant changes in the impregnant - graphite system occur in the 100° - 600°C temperature region. These results suggest that a slower heating rate during carbonization is likely to reduce spalling. Consequently, a more detailed investigation concerning the effect of the heating rate on the gas evolution and deposition of carbon in the pores is warranted. A more thorough understanding of these phenomena is certain to lead to a better knowledge of the causes of spalling.
- 4). Based on the results obtained thus far, a tentative mechanism for spalling is offered. The minimum permeability during carbonization (at 200°C) was found to be 2 to 3 orders of magnitude lower than the permeability found at the end of the carbonization (900°C). Upon subsequent impregnations and carbonizations resulting in permeabilities of 10^{-5} to 10^{-6} cm²/sec, it is likely that the permeability of the graphite is of the order of 10^{-8} to 10^{-9} cm²/sec during some portion of the carbonizing cycle. Therefore, any partially carbonized impregnant would evolve gases which could create a stress large enough to result in a spall or a crack in the graphite. During the carbonization cycle, the small pores inhibit the rate of release of gases formed in all of the pores. In the event that the gas release rates are slow

or completely inhibited, pressure pockets develop in the graphite body and with increasing temperature, the pressure in these volumes becomes great enough to cause a crack or spall in the graphite.

Application of Experimental Data

Based on the metallographic, gas evolution and permeability results, obtained, modifications in the manufacturing process of low permeability graphite from impregnated graphite was introduced. The temperature range for the gradual temperature rise (i.e. 4.2°C/hr) was extended from the 100° - 500°C range to the 100° - 600°C range and the carbonization cycle was extended to six days. The modified cycle was employed to make a prototype HTGR fuel element sleeve having a permeability of $8 \times 10^{-6} \text{ cm}^2/\text{sec}$ after graphitization. The heating cycle has been accepted, at the present time, as standard for a six day carbonization cycle. Furthermore, the same heating rate may be applied to the three and four day carbonization cycles.

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Appendix

Apparatus and experimental procedure to study the gas evolution characteristics of graphite - Specimens of impregnated HLM-85 (Great Lakes Carbon Co.) graphite approximately .45 inch by .85 inches and .375 inches thick were cut as sections from a 2.75 inch inside diameter and .375 inch wall thickness impregnated graphite tube.

Each of nine (9) Inconel capsules 3 inches long, 0.75 inches inside diameter and 0.060 inches wall thickness, was connected to a 0.085 inch inside diameter Inconel tube which was welded to a Hoke needle valve. Each graphite specimen was weighed prior to insertion in the respective capsules and the remaining end of each capsule was sealed by welding a metal cap to it. After each capsule was tested for vacuum ($\sim 10^{-6}$ mm.) tightness, the capsules were inserted into the nine holes (0.78 inches diameter, 3.75 inches deep, equally spaced on a 0.875 inch diameter) drilled in a circular type 304 stainless steel block 5 inches diameter and 4 inches long. The entire capsule assembly was then placed in the furnace and the capsules were connected to a common pyrex glass tube which in turn was connected to gas collecting flasks and a vacuum system. A photograph of the assembled apparatus appears in figure I. A pressure gauge was attached to the capsule which was eventually heated to 900°C.

At 100°C intervals, i.e. at 100°, 200°, 300°C etc., a gaseous sample was withdrawn by means of opening one of the valves connected to a capsule and allowing the gases to enter evacuated flasks. The capsule was then detached from the system and withdrawn from the furnace. The flasks containing the gases were disconnected from the system and replaced by similar flasks. The heating cycle continued in an uninterrupted manner and a similar procedure was followed at each of the temperatures at which a gaseous specimen was withdrawn. One of the gaseous samples was withdrawn from one of the capsules at 380°C due to the development of a slight leak from that capsule at approximately 375°C. Also in order to decrease the pressure at 400°C in the remaining capsules, a composite gaseous specimen was withdrawn from all of the remaining capsules at 400°C.

After the specimens were subjected to the heating cycles, the specimens were weighed and then cut and one portion was used for permeability measurements and the other portion was used for metallographic studies.

Figure I - Photograph of the apparatus used for the heat treatment cycle of the impregnated graphite and for collecting the evolved gases at various temperatures.