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1P-25A(PT-105-532-E)

AUTHOR

B. A. Ryan

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C-REACTOR GRAPHITE BURNOUT INTERIM REPORT -
1P-25A(PT-105-532-E)

By

B. A. Ryan
Nonmetallic Materials Development
Hanford Laboratories

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C-REACTOR GRAPHITE BURNOUT INTERIM REPORT -
1P-25A(PT-105-532-E)

INTRODUCTION

The oxidation of graphite in the Hanford reactors is of consequence since graphite burnout affects the strength of the moderator. As a means for indication of any highly oxidizing condition within the stack, containers or boats of small weighed samples of reactor-grade graphite are positioned along the length of an empty process channel in each reactor. The rate of oxidation of the monitoring samples, referred to as the burnout rate, is reported as percent weight loss per 1000 operating days (%/KOD). Currently the burnout rate limit is 2%/KOD (Process Standard C-040, September 1, 1959).

This document presents recent burnout data at C-reactor, under a program authorized by 1P-25-A(PT-105-532-E).

SUMMARY

1. Graphite monitoring samples in Channel 1960 have shown burnout rates significantly higher than at any of the other reactors; i.e., samples discharged in January, 1960, and in September, 1960, had 35% and 17%/KOD, respectively. Previously the highest burnout rate was about 5%/KOD in 3478-D.
2. The extent of the oxidation is believed to be limited primarily within channel 1960, although there is evidence to suggest that other areas of the stack were also oxidized.
3. Impurities, carried into channel 1960 by process water flowing over rusty gunbarrels, acted as catalysts and accounted for the extremely high burnout rates. The oxidizing agent was oxygen from inleakage of air into the reactor. This oxygen must also have contributed to the graphite damage of the stack in

general, but the amount of general oxidation has not been measured.

GRAPHITE MONITORING DATA

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Burnout Rates

From January, 1954, when the monitoring tests for C-reactor were initiated until March, 1958, there was no indication of excessive oxidation within the stack; however, a set of samples which was exposed in channel 1960 from March 4, 1958, to January 25, 1960, appeared to be severely damaged. During the discharge of the samples, the graphite sample carrier located nearest the centerline of the stack crumbled, and only two of the five samples were recovered. Photographs and autoradiographs of these two samples, which had an average burnout rate of 35%/KOD, are shown in Figure 1.

The next set of samples was discharged on September 8, 1960, after 178 operating days. The oxidation rates were not as high as previously, but still they were well above the limit, as can be seen from Table I. During the discharge of this test, several boats were observed to be partially coated with a red-brown substance that appeared to be rust. A subsequent gamma count of the material indicated it was iron.

Another set of samples was discharged from 1960-C on October 10, 1960, after 29 operating days. With the exception of one carrier, all the rates were well within limits, as shown in Table II.

The most recent monitoring test, which was discharged on February 26, 1961, after 115 operating days, showed burnout rates near the established limit of 2%/KOD. During this test two additional channels, 1889 and 2780, were charged with samples. To prevent the contamination of these samples during the charging operation, a process tube was used to line the inside of the gunbarrel. The tube extended a couple of feet in front of the Van Stone flange and the carriers

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were charged through it into the reactor. Through this method, no water from the front face or rust from the gunbarrel contaminated the samples during installation. However, when the boats were discharged, it was observed that water (perhaps 100 cc) poured from channels 1889 and 1960 as the first boats were pushed out. Furthermore, the samples in these boats were wet, and about 25 cc of water were poured from the boats as the samples were removed. Also, there was a ring of rusty material on the bottom of some of the carriers which was determined to be iron. The physical appearance of the carriers resembled those discharged on September 8, 1960, and on October 10, 1960.

Oxidation rates from this last monitoring test are summarized in Table III. Several observations are that: (1) The rates were very close to the established limit. (2) The rates from the same relative position in channels 1889 and 1960 were very nearly the same, while those in channel 2780 were considerably lower. (3) In each case the burnout rate reached a peak at boat #2, tapering off in each direction from this location. Undoubtedly the concentration of the oxidizing gas was greatest in front of the channel, but the graphite temperatures were low enough to result in less oxidation than in the vicinity of the first boat. Furthermore, by the time the gas stream reached the third carriers the concentration of the oxidizing species was markedly reduced as a result of reaction in the fore-part of the channel. (4) A weight gain in the fifth carriers in channels 1889 and 1960 indicated that all the oxidant was removed from the gas and that the reaction products disproportionated, plating out carbon on the samples.

Borescope Observations

To determine if excessive damage of monitoring samples was indicative of attack to the tube graphite, channel 1960 was inspected visually. About two

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weeks after the samples which had undergone a burnout rate of 35%/KOD were discharged (on January 25, 1960), the channel was borescoped. Although no photographs were taken, a vivid indication of the damage to the graphite is apparent from the following report:⁽³⁾

The channel had dark pitted areas which looked similar to the oxidized graphite burnout samples removed from this channel on a previous outage. The location of these pitted areas is as follows: at 13'5" a 1/2" wide streak begins and enlarges to encompass the entire channel at 15'11" for a distance of about 8". From this point to 24'10" the oxidized areas become patchy with the top of the channel being affected to a greater degree than the sides or bottom. It is estimated that 50 per cent of the channel surface area between 13'5" and 24'10" has been attacked. No estimate of the depth or penetration of the oxidation could be made.

Visual observation during a subsequent borescoping indicated graphite damage in vertical safety rod channels 22 and 34:⁽⁴⁾

On August 11, 1960 VSR channels 22, 34, and 35 were borescopedchannels 22 and 34 were found to have several round spots, darker than the normal graphite and of a pitted appearance. The spots were scattered throughout the central region of the channel and appear on the ends and sides of the filler blocks from near and far. These spots are similar in appearance to those observed on graphite burnout samples irradiated in C-Reactor a few months ago.

Hence, it is apparent from the borescoping observations that obvious oxidation of the graphite stack at 105-C has occurred, although the damage was most pronounced in channel 1960, for reasons which will be discussed in the following section.

DISCUSSION OF THE CAUSES FOR HIGH BURNOUT RATES

The Promotion of Catalyzed Oxidation by Impurities

It is a well-established fact that many impurities, eg., iron cobalt, nickel, copper and manganese, markedly influence the oxidation of graphite.⁽⁵⁾ Clark has demonstrated that impurities normally present in graphite which has not been subjected to gas purification increase the oxidation rate.⁽⁶⁾ These impurities

cause local, highly reactive sites which give rise to preferential oxidation, and the resulting oxidized graphite surface has the appearance of Swiss cheese.

The possibility that the samples discharged in January 1960 were prepared from a contaminated source of graphite is discounted since other samples from the same source showed normal weight losses during a regular monitoring test in 1880-KW at temperatures and fluxes that were substantially greater than at C-Reactor.

Photographs of the two samples that averaged a burnout rate of 35%/KOD are shown in Figure 1. They reveal the characteristic pitting of catalytic oxidation. The autoradiographs indicate the presence of impurities in pits on the samples. Gamma spectroscopy indicated the source of the activity was Fe⁵⁹. Scrapings from the craters on the samples, analyzed by emission spectroscopy, contained trace to moderate (10 to 50 ppm) amounts of iron and vanadium. In contrast, scrapings from the non-pitted area of the samples contained zero to less than trace amounts of these elements. (These concentrations compare with less than 10 ppm of iron and less than 1 ppm of vanadium for typical, "F" grade gas-purified graphite.)

As shown in Table IV, laboratory experiments substantiate the fact that iron and vanadium increase the oxidation rate of reactor-grade graphite. The oxidation rates of the treated samples are seen to be from two to seven times greater than the control sample. The erosion of the samples is shown photographically in Figure 2. Sample A, which was soaked in an iron and vanadium solution, illustrates that oxidation damage occurs to a greater extent on the leading or the upstream end; the control sample, B, gives no indication of selective oxidation, while the last sample, C, demonstrates that large mass removal is associated with the treated area.

From this evidence, it is believed contamination of the monitoring samples and the process channel graphite was by deposition of impurities either directly

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from the process water or redistributed by the water from gunbarrels or other components. As was mentioned earlier, rust appeared on several of the boats and water was pushed out of channels 1889 and 1960 during discharge of the last test. Furthermore, an entry in the Supervisor's Log⁽⁷⁾ indicated that there was water in 1960-C during the test discharged in January 1960, when burnout rates of 35%/KOD were observed:

When the tube (1960) was unbuttoned for sample discharge (it was found (that the) rear contained a short bayonet and shielding pieces only - no flange. Probably (there was) a good-sized gas leak. Water running into gunbarrel has produced large amounts of rust. Probably the gunbarrel will require replacement before channel is retubed.

The long-range effect of the deposition of catalysts is apparent. Once so affected, the graphite oxidation will continue at an abnormally high rate.

The Promotion of Oxidation By Air Inleakage

The presence of air in a reactor atmosphere has very deleterious effects upon the graphite through the reaction with oxygen. While the rate of graphite removal in the lowest temperature areas of the stack is probably negligible, the rate at temperatures above 400 C is extremely fast. In laboratory experiments at atmospheric pressure, for instance, the rate at 333 C is about 2%/KOD; at 475 C it is about $2.2 \times 10^3\%$ /KOD, and around 659 C the rate is $1.2 \times 10^5\%$ /KOD.^(8,9) Under the influence of ionizing radiation, these rates are two to five times faster.⁽⁹⁾

As already mentioned, the extremely severe burnout of monitoring samples from March 4, 1958 to January 25, 1960 was partially promoted by catalysis; however, there is evidence that excessive oxygen in the reactor atmosphere during the period March 4, 1958 to September 8, 1960, contributed to abnormally high burnout rates. To illustrate, referring to Figure 3, the oxygen content during this period was usually above the process limit of 0.05% (Process Standard B-010, December 14, 1960). While during the period October 10, 1960 to February 26, 1961 the oxygen content

remained within the limits, resulting in substantially lower burnout rates. (The values for the burnout rates in Figure 3 are means of the five samples in the highest oxidized boat.)

To further show the direct relationship between burnout rate and oxygen content, Figure 4 is included covering the period before 1958. Although there were occasional high oxygen values, the reactor generally operated within limits, resulting in low burnout rates. For comparison between reactors, the oxygen and burnout values for 105-DR are shown in Figure 5.⁽¹³⁾

As another indication of the damaging presence of oxygen in the gas atmosphere, oxidation rates vs position of monitoring samples in channel 1960 from January 25, 1960 to September 8, 1960 are plotted in Figure 6. Graphite temperatures from nearby thermocouples were available during this test and are shown in Table V. From the graph it is seen that the rate of graphite removal peaked very sharply in the upstream half of the stack, independent of the symmetrical distribution of the fast and thermal neutron flux and the rather symmetrical temperature distribution. (A description of the flux measurements appears in Appendix.) This indicates that a very large amount of the oxidant was removed from the gas stream in the 450-585° C area of the channel, preventing oxidation of the samples further downstream. Since CO₂ constituted a significant fraction of the coolant gas and since its reaction with graphite is insignificant below about 600° C, the extent of oxidation is deduced to have resulted from in-leakage of oxygen into the reactor atmosphere.

Thus, although the very high burnout rates observed in the 1960-C were partially the results of catalysis, the presence of oxygen from air in-leakage also seems an important contributing factor. This oxygen has a deleterious, although lesser, effect on the uncatalyzed regions of the stack.

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Table I

Burnout Rates in Channel 1960
From January 25, 1960 to September 8, 1960

<u>Carrier No.</u>	<u>Distance from Front of Graphite Stack to $\bar{C}$ of Carrier (ft)</u>	<u>*Burnout Rate (%/KOD)</u>
1	5.8	4.16 $\pm$ 0.56
2	7.6	9.81 $\pm$ 1.45
3	9.5	17.64 $\pm$ 6.19
4	11.2	5.12 $\pm$ 4.42
5	13.1	1.38 $\pm$ 0.09
6	14.9	0.77 $\pm$ 0.02
7	16.8	0.53 $\pm$ 0.04
8	18.6	0.25 $\pm$ 0.00
9	20.4	0.16 $\pm$ 0.01
10	22.3	0.004 $\pm$ 0.000

* Mean value for the five samples in each carrier.

Table II

Burnout Rates in Channel 1960
From September 8, 1960 to October 10, 1960

<u>Carrier No.</u>	<u>Distance from Front of Graphite Stack to $\bar{C}$ of Carrier (ft)</u>	<u>*Burnout Rate (%/KOD)</u>
1	3.92	0.17 $\pm$ 0.01
2	7.08	1.37 $\pm$ 0.07
3	10.3	5.40 $\pm$ 0.26
4	13.4	1.71 $\pm$ 0.44
5	16.6	0.52 $\pm$ 0.05
6	19.8	0.41 $\pm$ 0.05
7	22.9	0.05 $\pm$ 0.01
8	26.1	+0.07 $\pm$ 0.04 (gain)
9	29.2	+0.13 $\pm$ 0.05 (gain)
10	32.4	+1.41 $\pm$ 0.36 (gain)

* Mean value for the five samples in each carrier.

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Table III

Burnout Rates in 1889-C, 1960-C and 2780-C
From October 10, 1960 to February 26, 1961

<u>Channel</u>	<u>Boat No.</u>	<u>Distance from Front of Graphite Stack to $\bar{C}$ of Boat (ft)</u>	<u>*Burnout Rates (%/KOD)</u>
1889	1	4.0	2.12 ± 0.26
	2	8.8	2.35 ± 0.17
	3	13.7	0.29 ± 0.01
	4	18.5	0.23 ± 0.01
	5	23.3	$+0.11 \pm 0.01$ (gain)
1960	1	4.0	2.71 ± 0.10
	2	8.8	2.94 ± 1.42
	3	13.7	0.65 ± 0.01
	4	18.5	0.20 ± 0.01
	5	23.3	$+0.11 \pm 0.01$ (gain)
2780	1	4.0	0.12 ± 0.00
	2	8.8	0.60 ± 0.01
	3	13.7	0.36 ± 0.01
	4	18.5	0.18 ± 0.00
	5	23.3	0.03 ± 0.00

* The mean of the five samples is shown in this column.

Table IV

Oxidation Rates of Graphite
Treated with Iron and Vanadium*

<u>*Sample Number</u>	<u>Oxidation Rate (gm/gm hr)</u>	<u>Relative Rates</u>	<u>*Treatment History</u>
B	3.8×10^{-3}	1.0	Control sample - soaked overnight in distilled water.
A	8.9×10^{-3}	2.3	About 0.03 ppm of FeCl_3 and VOSO_4 added to sample by soaking overnight in a 0.001 M solution.
C	2.6×10^{-2}	6.8	About 3 ppm of FeCl_3 and VOSO_4 deposited along the top side of the sample.

* Tests were conducted in the laboratory by passing dry air (2 cfh) over the sample at 600 C, and they were continued to 10% weight loss of the sample.

+ Samples were prepared from TS-GBF type graphite and weighed 7 to 8 grams.

* Quantity of catalyst added to the samples was determined by the weight change after the treatment. In each case, the solvent or water was removed in vacuo before oxidation was started.

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Table V

Graphite Temperatures, °C, Near Channel 1960 from
January 25, 1960 to September 8, 1960 *

<u>Distance into</u> <u>Graphite Stack#</u> <u>(feet)</u>	<u>Stringer Located</u> <u>in Channel</u> <u>1561</u>	<u>Stringer Located</u> <u>in Channel</u> <u>2261</u>	<u>Stringer Located</u> <u>in Channel</u> <u>2267</u>
4	*38% > 450 11% > 475,495 max	70% > 400 11% > 420,440 max	39% > 350 10% > 375,485 max
10	45% > 525 16% > 550,570 max	--	31% > 500 17% > 550,585 max
20	50% > 400 15% > 425,470 max	70% > 400 19% > 450,470 max	69% > 400 20% > 450,470 max
25	70% > 165 24% > 185,220 max	--	30% > 165 13% > 175,225 max

* There were 178 operating days during this test.

The thermocouple positions relative to the burnout monitoring samples and the flux wires are shown in Figure 6.

* Refers to percentage of total operating days the thermocouple registered above the indicated temperature. From the graphite temperature records, it was impossible to obtain a time-weighted average since the readings were taken at irregular intervals.

FIGURE 1

Photographs and Autoradiographs of Samples with
Average Burnout Rate of 35% /KOD.

- A - About .03 ppm of FeCl_3 and VOSO_4 were added to sample of soaking overnight in a .001 M solution.
- B - This is the control sample; it was soaked overnight in distilled water.
- C - About 3 ppm of FeCl_3 and VOSO_4 were deposited along the top side of the sample.

FIGURE 2

Photograph Showing the Catalytic Effects of Trace Amounts of Iron and Vanadium.

FIGURE 3

Graph of Oxygen Content and Burnout Rates at C-Reactor from 1958 to 1961

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FIGURE 4

Graph of Oxygen Content and Burnout Rates at C-Reactor from 1955 to 1958

FIGURE 5

Graph of Oxygen Content and Burnout Rates at DR-Reactor from 1958 to 1961

Graph showing Oxidation Rate (%/1000 Operating Days) vs. Position in Graphite Stack (Inches). The graph includes data for Temperature (°C), Thermal Exposure (x 10²⁰ nrv), and Fast Exposure (x 10¹⁹ nrv). A vertical dashed line marks the 'Graphite Stack' center. A horizontal dashed line indicates 'Gas Flow' direction. The oxidation rate peaks around 40 inches from the left and right edges, with a minimum near the center. The temperature profile shows a sharp peak at the center, reaching 1800°C. The thermal and fast exposure profiles are relatively flat, around 12 and 6 respectively. The graph is labeled with 'T/C' at various positions along the x-axis.

FIGURE 6
Oxidation Rates and Temperature and Flux Data from January 25, 1960 to September 8, 1960

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The fast neutron exposures ($E > 1.0$ Mev) were obtained from the $\text{Ni}^{58} (n,p) \text{Co}^{58}$ reaction, while the thermal neutron exposures were based on the $\text{Co}^{59} (n,p) \text{Co}^{60}$ reaction. About 1/4" of nickel wire (99.995% pure and 0.020" diameter, obtained from Jarrell-Ash Co.) and about 1/4" Co-Al alloy wire (0.1% Co and 0.040" diameter, purchased from Sigmund Cohn Mfg. Co., Inc.) were used as the foils. These short pieces of wire were embedded in small discs of graphite which were placed between the graphite monitoring samples in carriers along the length of channel 1960 for irradiation.

After discharge of the carriers, the flux monitors were removed and counted as disintegrations per second per milligram (d/sec/mgm). Using equations presented by Morgan and Yoshikawa, values for the fast and thermal flux were calculated.^(14, 15) The familiar cosine curve for the profile of the flux along the length of the channel is seen in Figure 6. (The slight flattening at the peak is due to attenuation of the flux by control rod #11.)

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