FEB 5 1962

GENERAL ATOMIC DIVISION
GENERAL DYNAMICS CORPORATION

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MASTER

GAMD-2476

Category A

Copy No. 82

Processing of Graphite Specimens for High
Temperature Irradiation by Hanford

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Project No. 32.164
Contract No. AT(04-3)-314
U. S. ATOMIC ENERGY COMMISSION

August 9, 1961

628 801

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INTRODUCTION

A graphite irradiation experiment is being conducted jointly by General Atomic and Hanford. The objective is to determine the effect of various heat treatments and impregnation on the electrical resistivity, structural parameters, and irradiation effects. The pre-irradiation processing was performed at General Atomic, and the x-ray analysis and irradiation experiments will be done by Hanford. This report includes the work done at G. A. prior to shipping the samples to Hanford.

Material

The base graphite for the experiment was NCC's NPR-core, and GLCC's HLM-85. The HLM-85, impregnated with furfuryl alcohol, is of current interest to the HTGR as a sleeve material, and the NPR-core graphite is of interest to Hanford and G. A. as a stable in-pile structural, reflector, and moderator material. Both base graphites are manufactured from a petroleum coke filler bonded with coal tar pitch. The NPR-core graphite is manufactured from "needle coke" which has been recommended in the past as a stable graphite under high temperature irradiation. The HLM-85 has a fine pore structure and can be impregnated to obtain low gas permeability while the NPR-core graphite has an open structure.

A diagram of the sample shape and dimensions is shown in Figure 1.

Irradiation Conditions

The irradiation conditions will be specified by Hanford. However, it is understood the samples will be irradiated in uncontrolled capsules in the MTR core above 1000°C to relatively high nvt.

Experimental Work

The processing consisted of an initial heat treatment of the as manufactured base stock at 2900°C for about one-half hour, and subsequent impregnation, carbonization of the impregnant and final heat treatment to 2650 or 2900°C. The effect of heat treatments and impregnant coke on the electrical resistivity of the graphite samples is given in Table 1.

FIGURE 1 GRAPHITE IRRADIATION SAMPLES

TABLE 1.

EFFECT OF HEAT TREATMENTS AND IMPREGNANT
COKE ON ELECTRICAL RESISTIVITY

Number	Source	SAMPLE		Orientation with Respect to Extrusion Force	ELECTRICAL RESISTIVITY, μ OHM-CM					
		Type			As Manufactured	1st Treatment Heated to 2900- 2925°C in Argon for 25 min.	2nd Treatment 1st treatment + 1 impregna- tion with carbonization to ~900°C.	3rd Treatment 1st and 2nd treatments + a 2nd impreg- nation with carbonization to ~900°C.	4th Treatment 1st, 2nd and 3rd treatments + heating to 2650°C in Argon for 2 hours.	5th Treatment 1st, 2nd, and 3rd treatments + heating to 2900°C in Argon for 25 min.
HW-164-X1	HW-NCC	NPR		Perpendicular	1260	1135	----	----	----	----
HW-164-X2	HW-NCC	NPR		Perpendicular	1278	1125	1123	1089	----	----
HW-164-X3	HW-NCC	NPR		Perpendicular	1480	1315	1250	1195	1195	----
HW-164-X4	HW-NCC	NPR		Perpendicular	1410	1240	1189	1135	----	1075
HW-164-X10	HW-NCC	NPR		Perpendicular	1340	1225	----	----	----	----
HW-164-X11	HW-NCC	NPR		Perpendicular	1150	1074	1059	1036	----	----
HW-164-X12	HW-NCC	NPR		Perpendicular	1260	1153	1145	1110	1072	----
HW-164-X13	HW-NCC	NPR		Perpendicular	1180	1045	1022	995	----	943
HW-164-201X	HW-NCC	NPR		Parallel	630	584	----	----	----	----
HW-164-202X	HW-NCC	NPR		Parallel	630	590	572	555	----	----
HW-164-203X	HW-NCC	NPR		Parallel	641	613	586	544	536	----
HW-164-204X	HW-NCC	NPR		Parallel	659	623	594	548	----	523
HW-164-205X	HW-NCC	NPR		Parallel	682	630	----	----	----	----
HW-164-206X	HW-NCC	NPR		Parallel	727	666	638	604	----	----
HW-164-207X	HW-NCC	NPR		Parallel	767	691	663	633	603	----
HW-164-208X	HW-NCC	NPR		Parallel	570	512	507	486	----	476
GA-1494-7-1	GA-GLCC	HLM-85		Parallel	605	562	----	----	----	----
GA-1494-7-2	GA-GLCC	HLM-85		Parallel	593	555	539	529	----	----
GA-1494-7-3	GA-GLCC	HLM-85		Parallel	616	558	539	529	533	----
GA-1494-7-4	GA-GLCC	HLM-85		Parallel	591	531	539	529	----	529
GA-1494-7-5	GA-GLCC	HLM-85		Parallel	583	569	----	----	----	----
GA-1494-7-6	GA-GLCC	HLM-85		Parallel	611	586	581	565	----	----
GA-1494-7-7	GA-GLCC	HLM-85		Parallel	587	558	558	541	547	----
GA-1494-7-8	GA-GLCC	HLM-85		Parallel	605	576	565	548	----	559

Discussion of Results

An initial reduction in electrical resistivity was observed after heating the as manufactured material to 2900-2925°C. Subsequent heat treatments and impregnations reduced the resistivity only slightly. There was considerably more scatter in the electrical resistivity data of the NPR-core material as manufactured than for the HLM-85, and the scatter remained through subsequent treatments (See Figures 2 and 3). The HLM-85 is apparently more homogeneous than the NPR-core material. High final heat treatment (2900°C) did not improve on this characteristic.

The effect of carbon pickup on electrical resistivity of the impregnated samples is shown in Figures 4 and 5. The NPR-core graphite's open pore structure is apparent from the large carbon pick up (Figure 4) compared to that of HLM-85 (Figure 5). When the NPR-core samples were cut parallel to extrusion, there was a definite correlation of carbon pick up with resistivity; (see dotted lines in Figure 4) the material of higher resistivity soaked up the most carbon. This correlation was not as pronounced for samples cut perpendicular to extrusion. The correlation was not observed in HLM-85 samples, however, the resistivity values were quite close together in all HLM-85 samples. Thus, the resistivity measurements may not have been sensitive enough to show the effect.

X-ray diffraction samples were scraped from each sample after each processing step. The x-ray samples, along with the irradiation samples, were shipped to Hanford.

FIGURE 2 ELECTRICAL RESISTIVITY VERSUS TREATMENTS OF NPR-CORE GRAPHITE

FIGURE 3 ELECTRICAL RESISTIVITY VERSUS TREATMENTS OF HLM-85 GRAPHITE
PARALLEL TO EXTRUSION

FIGURE 4 EFFECT OF CARBON PICKUP DURING IMPREGNATION ON ELECTRICAL RESISTIVITY OF NPR-CORE GRAPHITE

FIGURE 5 EFFECT OF CARBON PICKUP DURING IMPREGNATION OF
ELECTRICAL RESISTIVITY OF HLM-85 GRAPHITE