AEC RESEARCH AND
DEVELOPMENT REPORT

HW-82436

GRAPHITE BURNOUT MONITORING

PART I

MONITOR PREPARATION AND HANDLING TECHNIQUES

B. A. RYAN

JUNE 1964

HANFORD LABORATORIES

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

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HW-82436

UC-25, Metals, Ceramics,
and Materials
(TID-4500, 32nd Ed.)

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By

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Materials Research and Service
Reactor and Fuels Laboratory

Hanford Laboratories

June 1964

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

Work performed under Contract No. AT(45-1)-1350 between
the Atomic Energy Commission and General Electric Company

Printed by/for the U. S. Atomic Energy Commission

Printed in USA. Price 75 cents. Available from the Office
of Technical Services, Department of Commerce,
Washington 25, D. C.

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INTRODUCTION

Small graphite samples have been used for a number of years to monitor graphite oxidation (burnout) in the Hanford reactors. Such monitors serve a very useful purpose as they provide a warning of the leakage of air into the reactors' gas systems and allow appropriate corrective steps to be taken before damage occurs to the graphite stacks. The monitors have proven helpful in assessing the effects of production tests, such as changes in temperature, gas composition and gas flow rates; also they have provided information on the depth of burnout from the graphite-O_2, graphite-H_2O, and graphite-CO_2 reactions.

It has long been recognized that a consistent method of preparing and handling monitors was needed. Occasionally large variations occur on monitors in adjacent test positions, and it is important to know that these are not variations within the monitors themselves. Monitors are sometimes returned to the laboratory with burned-out pits or rust-colored deposits, and it is helpful to know if this is indigenous to the monitors or attributable to reactor operation. Certain procedures for sample preparation and handling have been devised, and these are described in this report. The results of recent monitoring tests are reported elsewhere.[1]

SUMMARY AND CONCLUSIONS

Standard dimensions for graphite oxidation monitors are 0.43 in. diam x 3.9 in. long. The monitors are machined from six well-identified bars of TSGBF graphite. All the monitors are cut parallel to the extrusion axis of the bars. Results from ashing representative samples from the six bars of TSGBF indicate that the monitors possess equivalent chemical purity. Two standard designs are used for the monitor carriers, one providing information on the depth of oxidation.

All handling operations are with tongs or rubber gloves, and the monitors are always stored in a desiccator or in cardboard tubes to insure against contamination. Handling losses are insignificant. Desiccation before the initial and final weighing is considered important.

Relative chemical reactivities toward air were determined for a number of graphites (KS, KC, CS, CSF, SGBF, CSGBF, and TSGBF) to allow comparisons between the various graphites in the Hanford reactors.

EXPERIMENTAL RESULTS AND DISCUSSION

Monitors and Monitor Carriers

The usual assembly for determining the extent of oxidation consists of a number of cylindrical samples in a perforated, hollow, cylindrical carrier which has an inside diameter considerably larger than the sample diameter.

The size and configuration of these components are dictated by the size of the reactor channel, the magnitude of an acceptable surface-to-weight ratio, and the necessity of maintaining an adequate flow of gas through the carriers and around the samples. The standard monitor size is 0.43 in. diam x 3.9 in. long; the carrier size is 1 1/8 in. OD x 7/8 in. ID x 20 1/2 in. long (Figure 1). This choice of carrier diameter permits 18 carriers to be fabricated from a single graphite bar and the choice of length permits storage in standard cardboard mailing tubes. The mailing tubes keep the assemblies clean before insertion into the reactor and prevent spread of radioactive contamination during shipment after discharge.

It is difficult to estimate the depth of oxidation into the large bars of moderator graphite from burnout data. This is an important problem since the integrity of the stack depends upon adequate compressive strength in the graphite. For a given amount of oxidation the strength is reduced more quickly when the burnout occurs uniformly throughout the bars than when it occurs just at the geometrical surfaces. There are empirical mathematical equations to predict the depth of oxidation; but, under reactor conditions where the variables are uncertain or at least vary from area to area, the equation is difficult to use. To obtain empirical data on the extent of oxidation below the geometrical surface, a variation of the standard carrier was devised which contains two separate compartments. One compartment permits the reactor gas to circulate freely around the monitors, and the other shields the monitors by a wall of graphite 3/8 in. thick through which

the oxidizing gas must diffuse to reach the monitors. A photograph of the open-closed type carrier is also shown in Figure 1.

In addition to the standard and the open-closed carriers, a third style of carrier has been constructed. It consists of a 1/4 in. diam solid cylinder, four progressively larger hollow cylinders and an outside shell. These pieces fit snugly inside one another and provide a technique for measuring the depth of oxidation through six layers of graphite. The pieces are enclosed in a graphite shell by a screw cap, and the upstream end contains three flat discs held in place by a graphite screw. The separate pieces are weighted before and after each monitoring period. Figure 2 is a sketch of this carrier.

Storage and Handling of Monitors Many impurities act as catalysts in the oxidation of graphite. Therefore, constant care is observed in handling and storing the monitors. The machining is done with equipment assigned exclusively for use with graphite, and all handling of the finished monitors is with rubber-tipped tongs or gloves. The monitors and carriers are stored in covered containers to prevent surface contamination. The only time that the monitors are exposed to possible foreign contaminants is during their actual residence in the monitoring facility or during the charge-discharge operation. During this latter step, care is exercised to keep water from the graphite.

Charging of the loaded carriers into process channels from which the process tube has been removed occurs during reactor shutdown. Formerly three carriers were positioned to obtain burnout rates from the front, central and rear portion of the stack; now, however, at least ten carriers per channel are considered necessary. After a nominal exposure of 3 mo, the carriers are pushed to the rear of the process channel by aluminum push-poles inserted from the front face of the reactor. The monitors are recovered on the rear elevator and are placed in clean cartons inside lead shipping casks. Until recently, the carriers were discarded, but now they are reused as many times as the radiation level allows, making it possible to obtain burnout rates on the large carriers themselves as well as to save material and labor costs. Occasionally the monitors and carriers are returned from the reactor with a rust-colored surface deposit which is

primarily iron and vanadium (see pp. 6-8 of Reference 2). From the position of the deposit, it appears that the graphite had passed through a puddle of rusty water. Whenever contaminated graphite such as this is discovered, it is retired from the monitoring program.

The monitors are held in a vacuum desiccator for 24 hr to remove moisture before each weighing, and they are weighed before and after each test. The burnout rate is calculated from the percent of weight change and the operating exposure, i.e., percent per 1000 operating days. The data are normally presented as burnout profiles in which the burnout rate is plotted against position along the graphite channel.

Evaluation of Handling Losses A test was performed to determine the losses encountered in transporting the monitors between the laboratory and the reactor and in transferring the monitors from the carriers to the shipping casks. For this purpose, a normal charge of ten carriers was prepared, carried to the reactor, pushed through one of the monitoring channels, retrieved on the rear face where the monitors were removed from the carriers and then transferred to ice-cream cartons in a lead cask. The cask and monitors were brought back to the laboratory. Next the monitors were weighed and then placed in a vacuum desiccator for 24 hr and finally weighed again. In all these operations, handling was performed in a routine manner. The results are shown in Table I.

The average and standard deviation from handling losses were 0.3 ± 0.2 mg; these values from dessiccation were 0.1 ± 0.2 mg. From these results, it can be concluded that even if the monitors were used for only a small portion of a normal 3 mo period, 10 days for example, the contribution to the burnout rate from handling errors would amount to only 0.3% per 1000 operating days. Therefore, this source of error is not significant. Although the average change in weight by desiccation appears insignificant for this particular test, desiccation is still employed because there is the possibility that the monitors can be showered by water during the discharging operations.

TABLE I

WEIGHT LOSSES ENCOUNTERED IN HANDLING AND DESICCATING BURNOUT MONITORS

Carrier Number and Type	Monitor Number	Original Weight, g	Weight after Charge-Discharge, g	Weight after Desiccation, g	Handling Losses mg	Desiccation Losses, mg
1 (Standard)	177-911	15.5540	15.5532	15.5531	0.8	0.1
	-912	15.2784	15.2773	15.2775	1.1	+0.2
	-913	15.2207	15.2205	15.2203	0.2	0.2
	-914	15.2222	15.2219	15.2216	0.3	0.3
	-915	15.4030	15.4026	15.4025	0.4	0.1
2 (Standard)	177-902	15.5959	15.5954	15.5952	0.5	0.2
	-903	15.5834	15.5832	15.5830	0.2	0.2
	-904	14.5973	15.5970	15.5970	0.3	None
	-905	15.5360	15.5359	15.5357	0.1	0.2
	-906	15.6080	15.6078	15.6076	0.2	0.2
3 (Standard)	177-753	15.8825	15.8821	15.8822	0.4	+0.1
	-754	15.9165	15.9162	15.9160	0.3	0.2
	-755	15.9870	15.9865	15.9865	0.5	None
	-756	16.0000	16.9999	16.9994	0.1	0.5
	-901	15.5846	15.5843	15.5841	0.3	0.2
4 (Open-Closed)	177-745	15.7088	15.7087	15.7085	0.1	0.2
	-746	16.0562	16.0560	16.0559	0.2	0.1
	-751	15.9498	15.9495	15.9494	0.3	0.1
	-752	15.8109	15.8105	15.8103	0.4	0.2
5 (Standard)	177-656	15.9209	15.9205	15.9203	0.4	0.2
	-655	15.9421	15.9417	15.9418	0.4	+0.1
	-654	15.9122	15.9119	15.9120	0.3	+0.1
	-743	15.6405	15.6397	15.6398	0.8	+0.1
	-744	15.6265	15.6266	15.6262	+0.1	0.4
6 (Open-Closed)	177-732	15.5264	15.5263	15.5264	0.1	+0.1
	-733	15.6778	15.6775	15.6775	0.3	None
	-734	15.6964	15.6961	15.6961	0.3	None
	-735	15.8725	15.8717	15.8715	0.8	0.2
7 (Standard)	177-723	15.6516	15.6512	15.6508	0.4	0.4
	-724	15.6074	15.6072	15.6070	0.2	0.2
	-725	15.6555	15.6550	15.6548	0.5	0.2
	-726	16.0626	16.0623	16.0623	0.3	None
	-731	15.9814	15.9813	15.9813	0.1	None
8 (Open-Closed)	177-715	15.8300	15.8299	15.8296	0.1	0.3
	-716	15.9967	15.9959	15.9963	0.8	+0.4
	-721	15.8948	15.8945	15.8047	0.3	+0.2
	-722	15.5012	15.5008	15.5006	0.4	0.2
9 (Standard)	177-706	16.0249	16.0244	16.0243	0.5	0.1
	-711	15.9665	15.9663	15.9660	0.2	0.3
	-712	15.8009	15.8010	15.8005	+0.1	0.5
	-713	15.6838	15.6836	15.6847	0.2	+1.1
	-714	15.6757	15.6753	15.6750	0.4	0.3
10 (Standard)	177-701	15.9378	15.9378	15.9377	None	0.1
	-702	15.8925	15.8922	15.8920	0.3	0.2
	-703	15.9292	15.9290	15.9287	0.2	0.3
	-704	15.9135	15.9130	15.9129	0.5	0.1
	-705	15.9239	15.9237	15.9235	0.2	0.2

Selection and Preparation of Burnout Monitors from Large Graphite Bars

It has been customary to use monitors made from the same type of graphite that had been used in the construction of the reactors. This method was recently changed because of the problems involved in record keeping and storing such a large number of specimens and because of the extra radiation exposure from less pure graphite. The current procedure is to use only one graphite, TSGBF, for all the graphite burnout monitors, and then by adjustment of the burnout rates measured from this material, it is possible to calculate a rate for other grades of graphite.

The accelerating effects of catalytic impurities upon chemical reactions can be complicated by uneven distribution within a large graphite bar even after the manufacturing processes.[3] For this reason it is possible to observe variations in the chemical reactivity of monitors cut from different areas of the same large bar. To insure against this source of error, six TSGBF bars from which monitors were to be prepared were carefully characterized by ashing representative sections from the bars and examining for atypical impurity patterns. The ashing method has been employed on other occasions and was shown to be a convenient method for examining the impurity distribution in large pieces of graphite. The graphite in areas containing high concentration of ash was not made into monitors.

The manner in which the TSGBF bars were sampled for ash patterns is illustrated in Figure 3. The dimensions of the original extruded bars were about 4 x 4 x 50 in., but the bars were trimmed uniformly to final dimensions of 3.75 x 3.75 x 49.15 in. Six sections about 0.6 in. thick were removed from each of the six bars; these sections are indicated in the diagram as A, B, C, D, E, and F. The sections were placed on ceramic tiles and slowly oxidized in a muffle furnace at about 750 C in stagnant air until completely ashed. (Occasionally one of the ceramic tiles, Section E of Figure 4 for example, cracked as a result of being removed from the furnace before cooling sufficiently.) Photographs of the ash patterns from the six TSGBF graphite bars can be seen in Figures 4 thru 9. One section of Bar No. 175 in Figure 4 was especially dense with impurities. Both ends of Bar No. 176, especially along the outside edges and at the corners, had large amounts of ash as shown

in Figure 5. The center of section E shows that there was some interaction of the impurities with the material in the tile.

Bar No. 177 was quite free of ash except along the bottom edge of Sections B, E, and F, as shown in Figure 6. Only one end of Bar No. 178 contained much ash, as shown in Figure 7.

After several days' use, the new furnace developed enough oxidized scale on the top heating elements to partially mask the graphite ash pattern, as shown in Sections E and F in Figure 8. Images of the heating elements can be seen as parallel, vertical lines in Section F.

Contamination from the oxidized heating elements in the furnace grew progressively worse as shown in Sections A and B of Bar No. 180 in Figure 9. To correct this problem, the heating elements were reoriented so that the bare metal was not exposed to the working chamber of the furnace.

Photographs of the ash patterns of the six TSGBF bars showed these general features:

- The ends of the bars contained greater amounts of ash than the central regions, and one end of the bars was usually richer in ash content than the other end.
- The outside edges and corners frequently contained greater concentrations of ash than the centers of the bars.
- Occasionally the central region of an end section contained a substance that interacted with the ceramic tile. This observation suggests that the chemical composition of the ash in the bars was not uniform; however, no confirming chemical analyses were performed. The possibility that localized hot-spots could have promoted this interaction can be discredited since the temperature of the furnace was controlled within ±6 C.

The ashing results showed that certain areas of the six graphite bars contained high concentrations of impurities which probably could enhance the chemical reactivity of burnout monitors machined from these areas. Therefore, to eliminate this possibility, the areas with high

concentrations of impurities were rejected, and burnout monitors were machined only from those areas in the bars containing the least amount of impurities.

In addition to characterization of the six TSGBF bars by ash patterns, apparent density and DIH (delta in-hour) values were also determined. The results are shown in Table II. DIH values determined in the Hanford 305 Test Reactor are a measure of very small concentrations of impurities that depress the flux in the test reactor. The relation between DIH values and the average absorption cross section per carbon atom for thermal neutrons (2200 m/sec) in millibarn units is:[4]

$$\sigma_{a\ (2200\ m/sec)} = 4.47 \pm 0.04 - (0.733\ DIH \pm 0.03).$$

It is expected that any influence upon the chemical reactivity caused by the slight variations in density and DIH values would be far outweighed by the influence of highly localized impurities; and, since the graphite near these impurity centers was discarded, all the monitors are believed to possess nearly uniform chemical reactivity.

TABLE II

IDENTIFICATION OF THE SIX BARS
OF TSGBF GRAPHITE USED FOR BURNOUT MONITORS

Bar	Heat	Apparent Density, g/cm^3	DIH Values	σ_a (2200 m/sec), mb
175	1251	1.72	1.062	3.69 ± 0.01
176	1339	1.71	0.980	3.75 ± 0.01
177	1253	1.71	1.026	3.72 ± 0.01
178	1339	1.69	0.972	3.76 ± 0.01
179	1339	1.69	0.942	3.78 ± 0.01
180	1339	1.70	0.954	3.77 ± 0.01

The 4 in. long slabs between the ashing sections, shown as I, II, etc., in Figure 3, were sawed into 36 rectangular samples, all parallel to the extrusion axis of the bars. These samples were numbered in the

manner shown in Figure 3. Finally the rectangular samples were cut into solid cylindrical burnout monitors with dimensions of 0.43 in. OD x 3.9 in. long.

From the six graphite bars 2160 burnout monitors were obtained. After discarding the ones located near high impurity centers, there remained an adequate supply for several years' use in the burnout monitoring program, especially since most monitors can be reused several times.

Comparison of Chemical Reactivity in Several Graphites

The chemical reactivity of various graphites differs somewhat due to the nature of the starting materials (coke and binder), the density, pore-size distribution, treatment temperature, and degree of purification. Since the standard monitors were made from only one graphite, TSGBF, it was necessary to correlate the chemical reactivity of this material with the various graphites used in the construction of the reactors. Therefore, a number of graphites (KS, KC, CS, CSF, SGBF, CSGBF, and TSGBF) were oxidized in air under constant conditions of temperature, gas composition, and gas flow-rate.

Samples of the various graphites were machined into solid cylinders 1/4 in. diam x 2 in. long. The samples were removed from the same region within each bar. More samples from several bars would be required to be sure that oxidation rates are representative of a particular grade.

The sample carriers were 1 3/4 in. diam x 6 1/4 in. long cylinders with seven 11/32 in. channels drilled all the way through. Each channel was used for a different graphite. Three samples, designated as upstream, middle, and downstream, were positioned in the channels. A carrier and some of the samples are shown before and after oxidation in Figure 10.

The carrier and its samples were positioned equidistant from the walls and the ends of a 5 in. diam x 36 in. long quartz combustion tube. The oxidation temperature was controlled by a thermocouple located between the tube and the furnace, and the temperature of the carrier was followed by thermocouples embedded at three positions along the length of the carrier. Since the difference in the diameters of the channels and the samples was

uniform and since the carrier was centered in the combustion tube, the flow of gas was considered to be uniform over all the samples.

The carrier and samples were heated to ~ 758 C, and then a flow of air (2 ft^3/hr) was started through the combustion tube. From the difference between the initial and final weights of the samples and the duration of the experiment (4 hr for the first experiment and about 30 hr for the others), the oxidation rate was calculated and expressed as $(g)(g^{-1})(hr^{-1})$. The results of four separate runs are shown in Figure 11, where the units on the coordinate axis are arbitrary.

New carriers and samples were used for each experiment due to the considerable loss of weight, as indicated in Figure 10. From the figure it can be seen that the greatest reaction occurred on the upstream ends where the gas stream impinged directly onto the carrier and samples. The middle samples received the least oxidation, an effect due to the inhibition of the reaction product CO or to the gas flow-pattern. There was a large excess of oxygen; it is estimated that enough oxygen passed through the combustion tube during the 30 hr runs to completely oxidize 20 sets of carriers and samples.

Although there was overlapping of the rates, the general trend, particularly with the upstream samples, showed increased chemical reactivity in going from the very pure SGBF to the less pure KS graphite. Some of the spread of data within individual graphite types was the result of handling losses. These were difficult to control because of powdery ends of the highly oxidized samples. As expected the results showed that the thermally-purified graphites (CS, KC, and KS) oxidized more rapidly than the gas purified graphites (SGBF, CSF, CSGBF, and TSGBF). The line drawn through the upstream samples is somewhat arbitrary, but the general trend is clear. The KS graphite oxidized approximately 1.5 times faster than TSGBF graphite.

ACKNOWLEDGEMENTS

The author acknowledges the assistance of S. E. Nichols for much of the laboratory work reported in this paper.

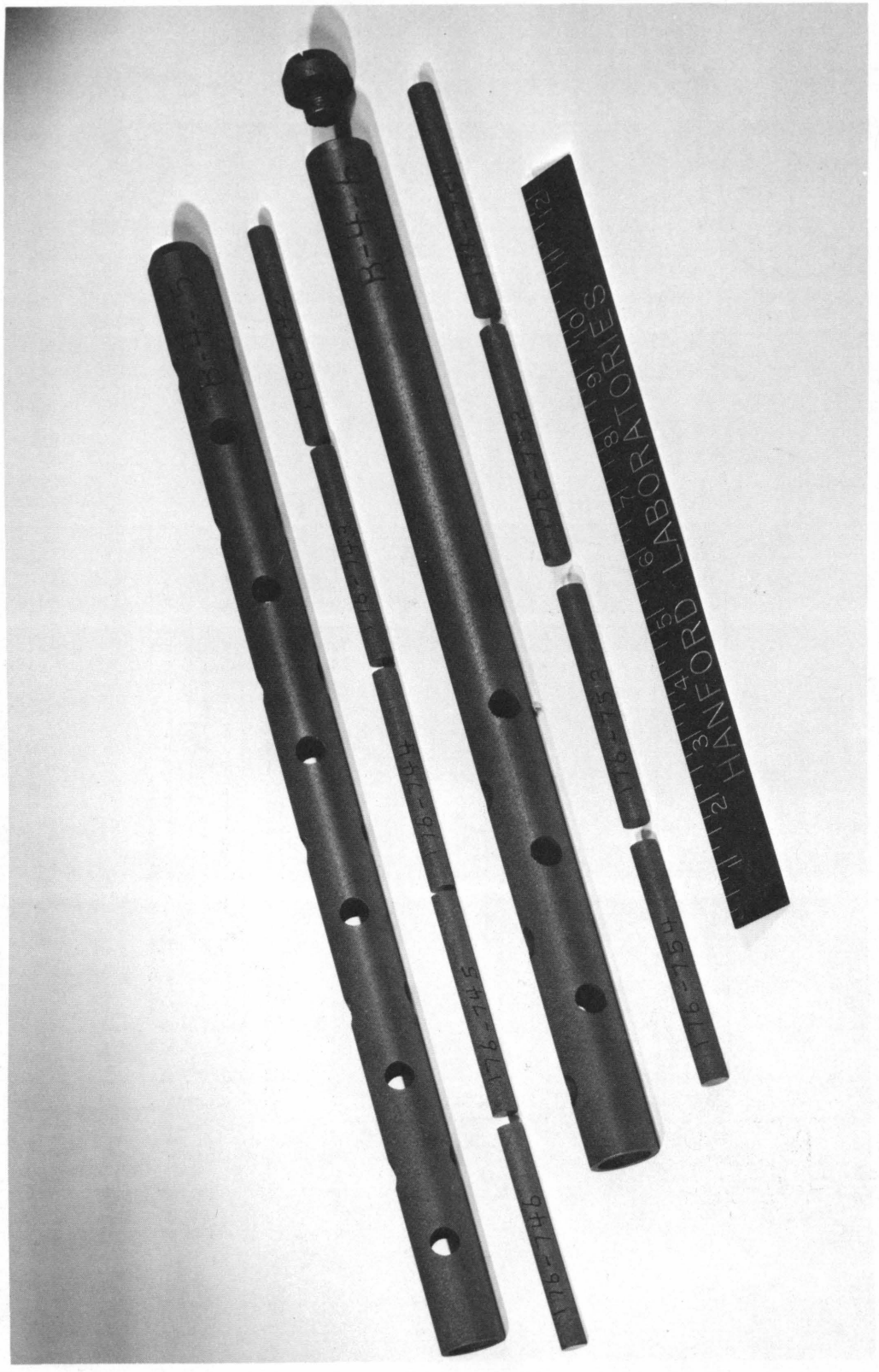

FIGURE 1

Monitors and Two Types of Carriers

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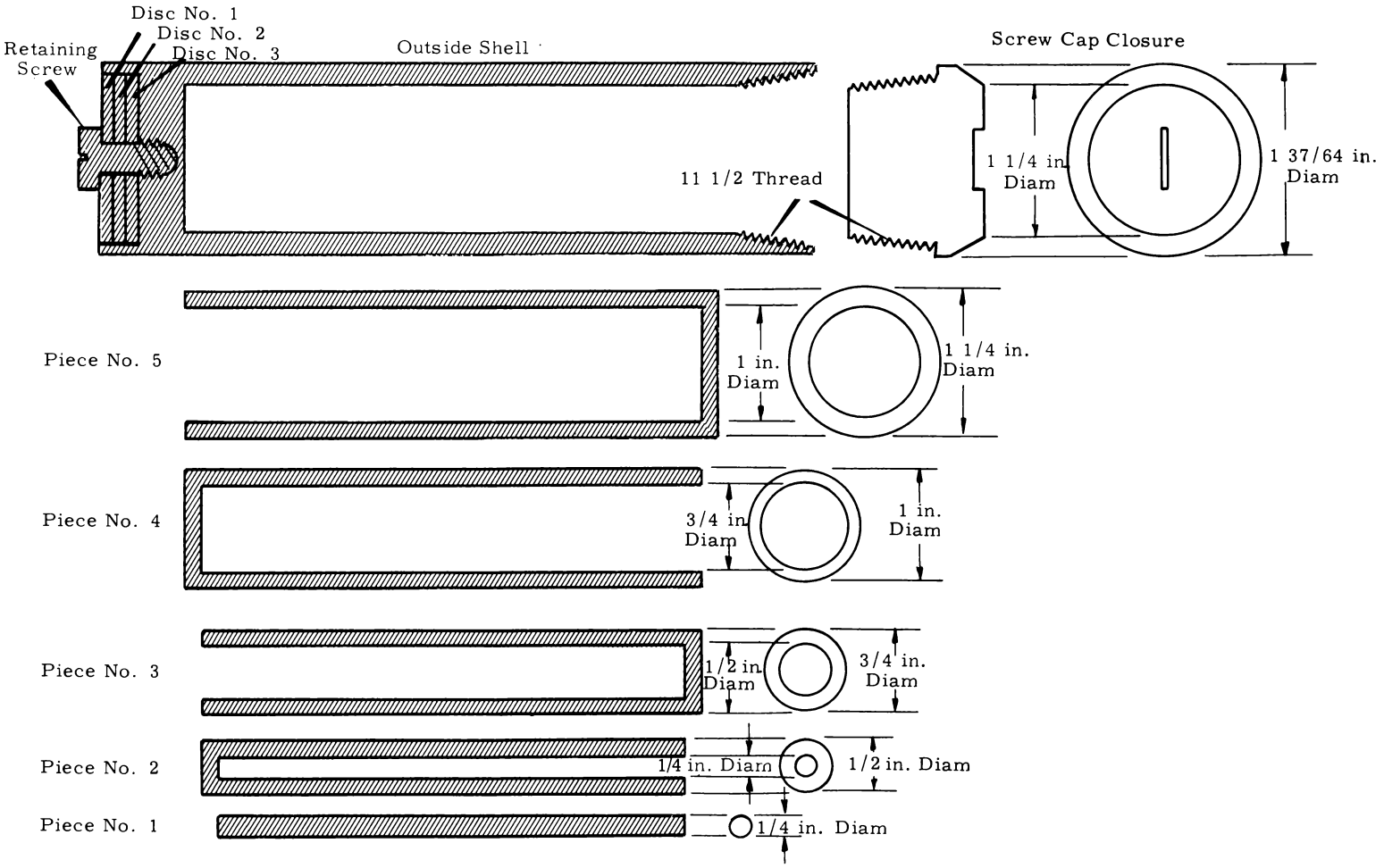

FIGURE 2

Diagram of Special Graphite Burnout Monitoring Assembly

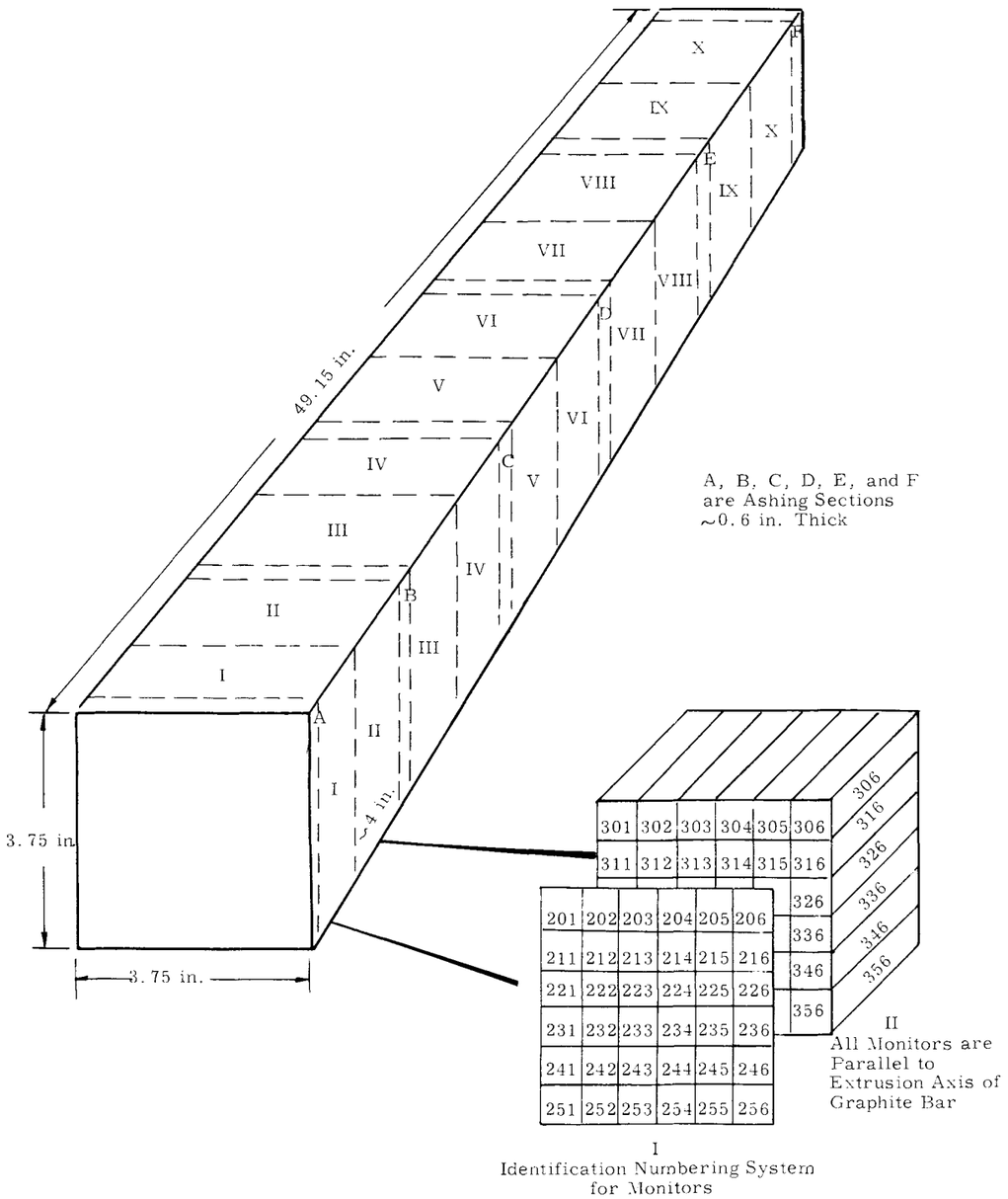

FIGURE 3

Diagram of Graphite Bar Showing Areas for Obtaining Ash Patterns and Identification Method of Burnout Monitors

AEC-GE RICHLAND, WASH.

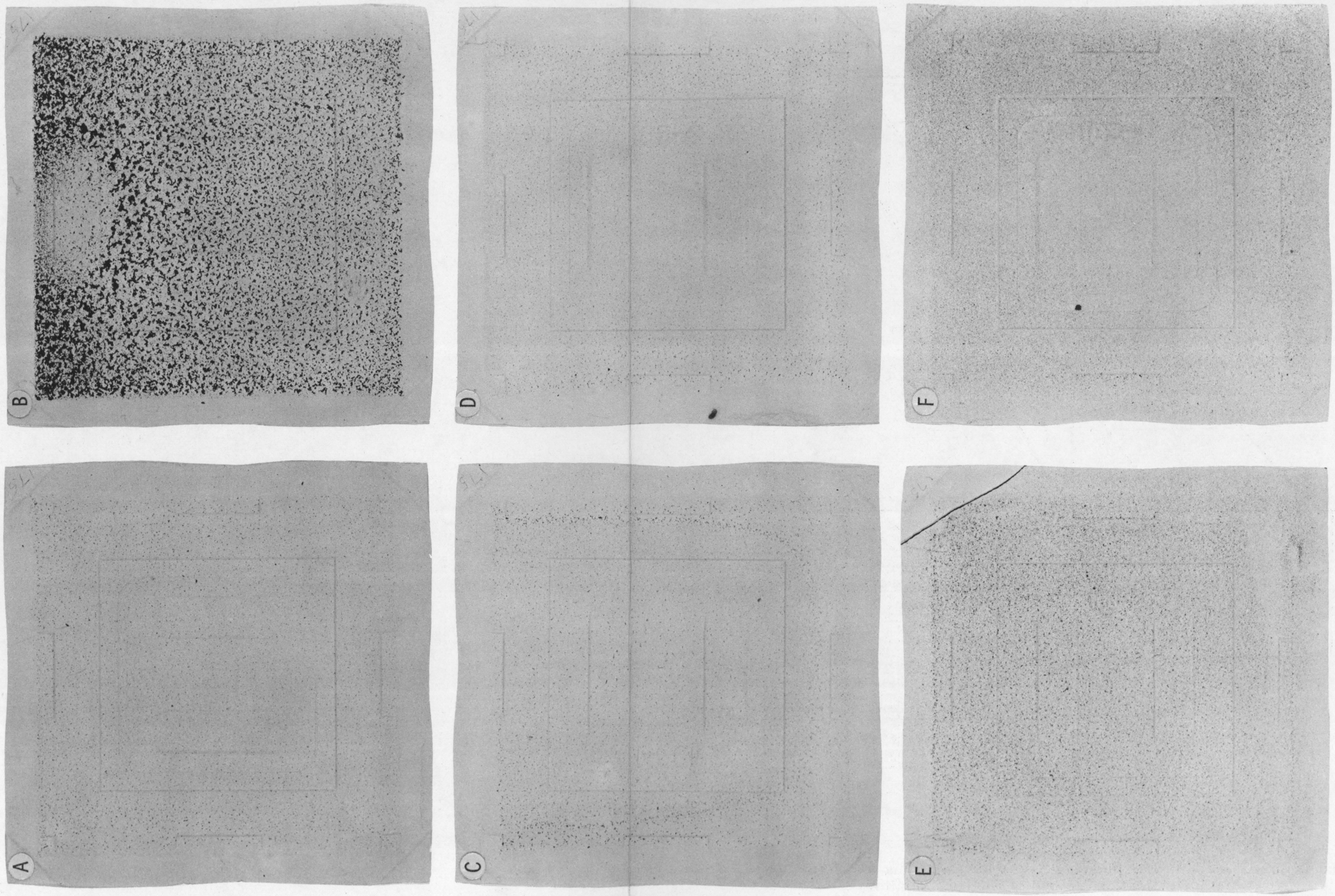

FIGURE 4

Ash Patterns from Bar No. 175

AEC-GE RICHLAND, WASH.

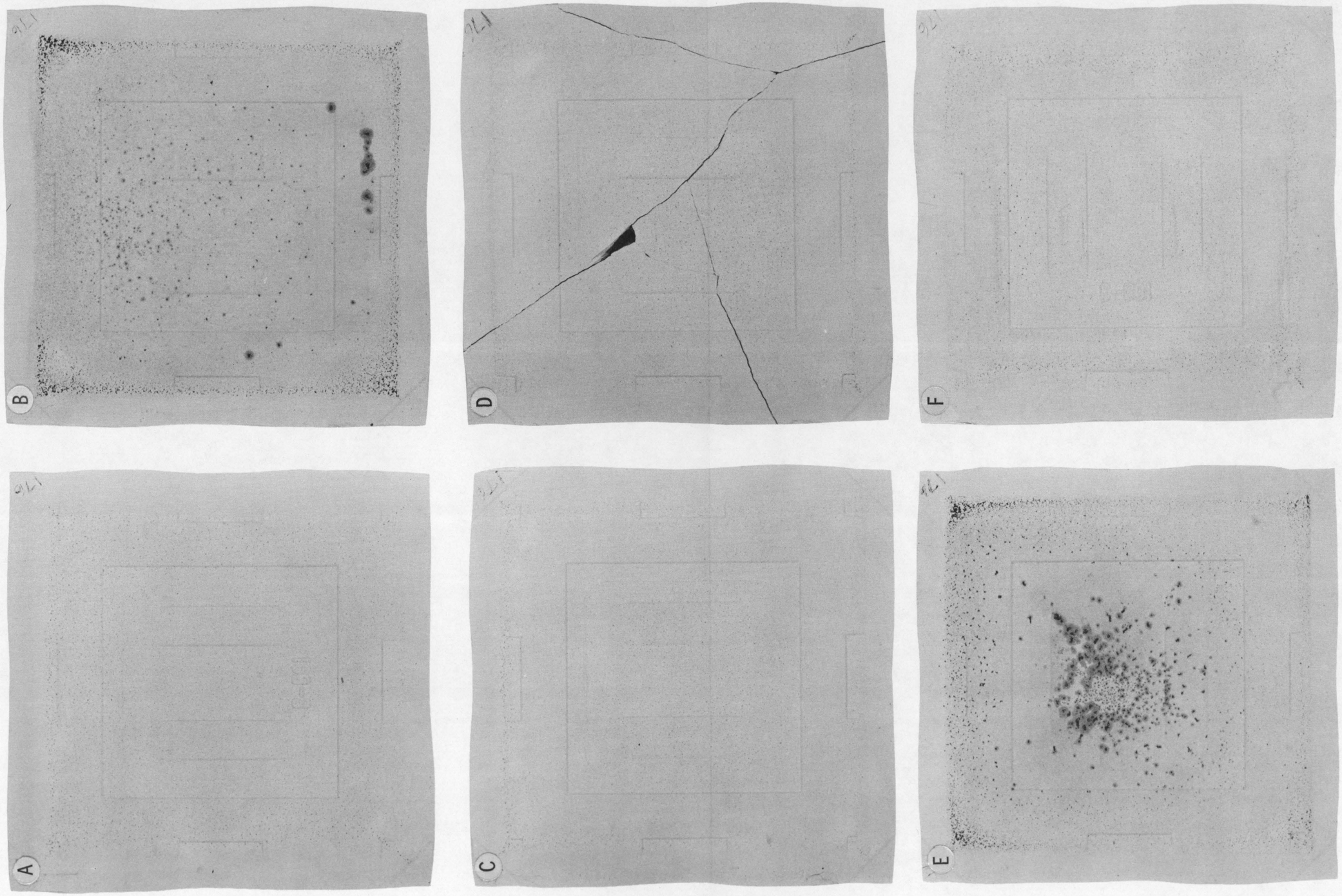

FIGURE 5
Ash Patterns from Bar No. 176

AEC-GE RICHLAND, WASH.

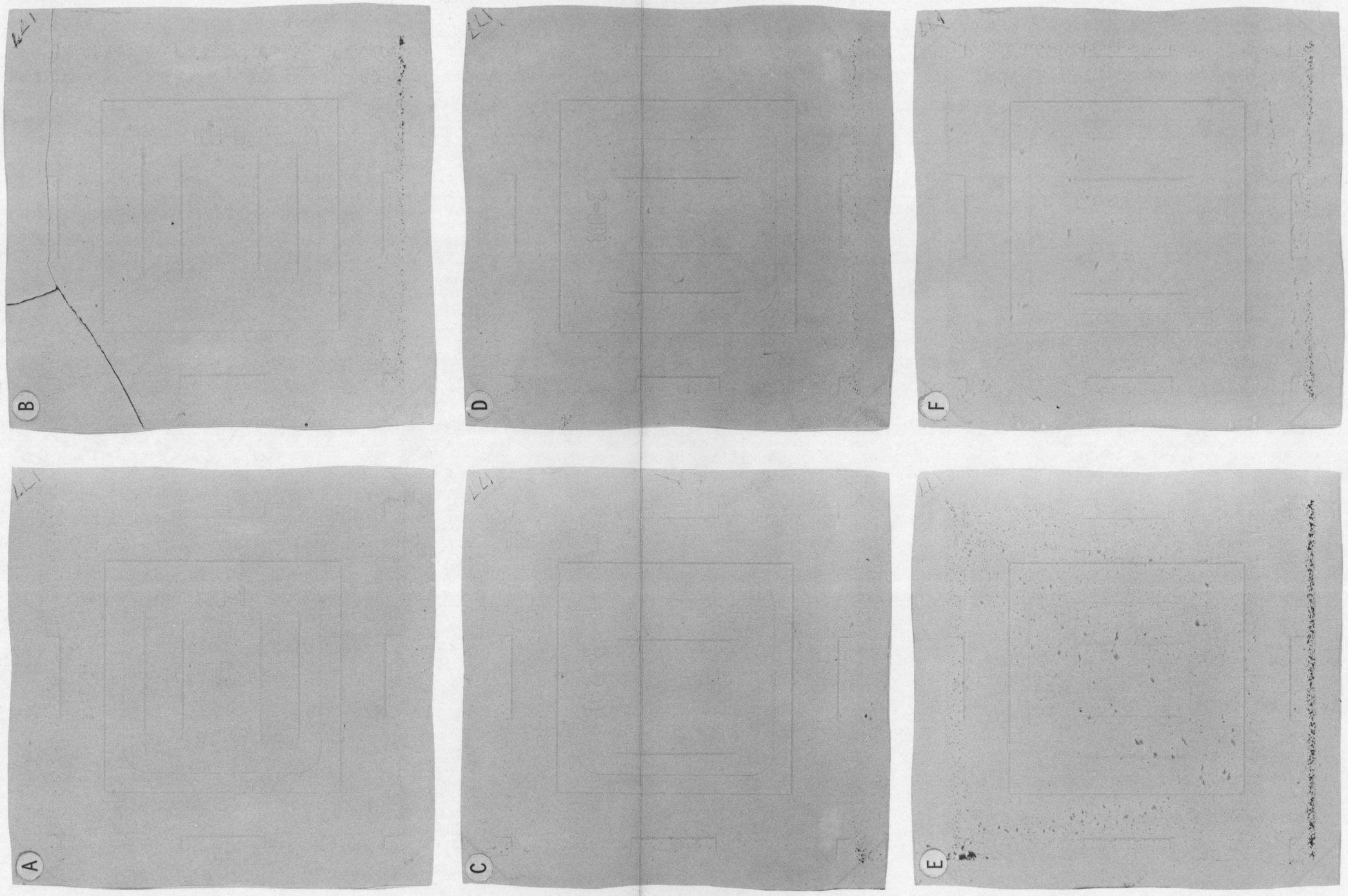

FIGURE 6
Ash Patterns from Bar No. 177

AEC-GE RICHLAND, WASH.

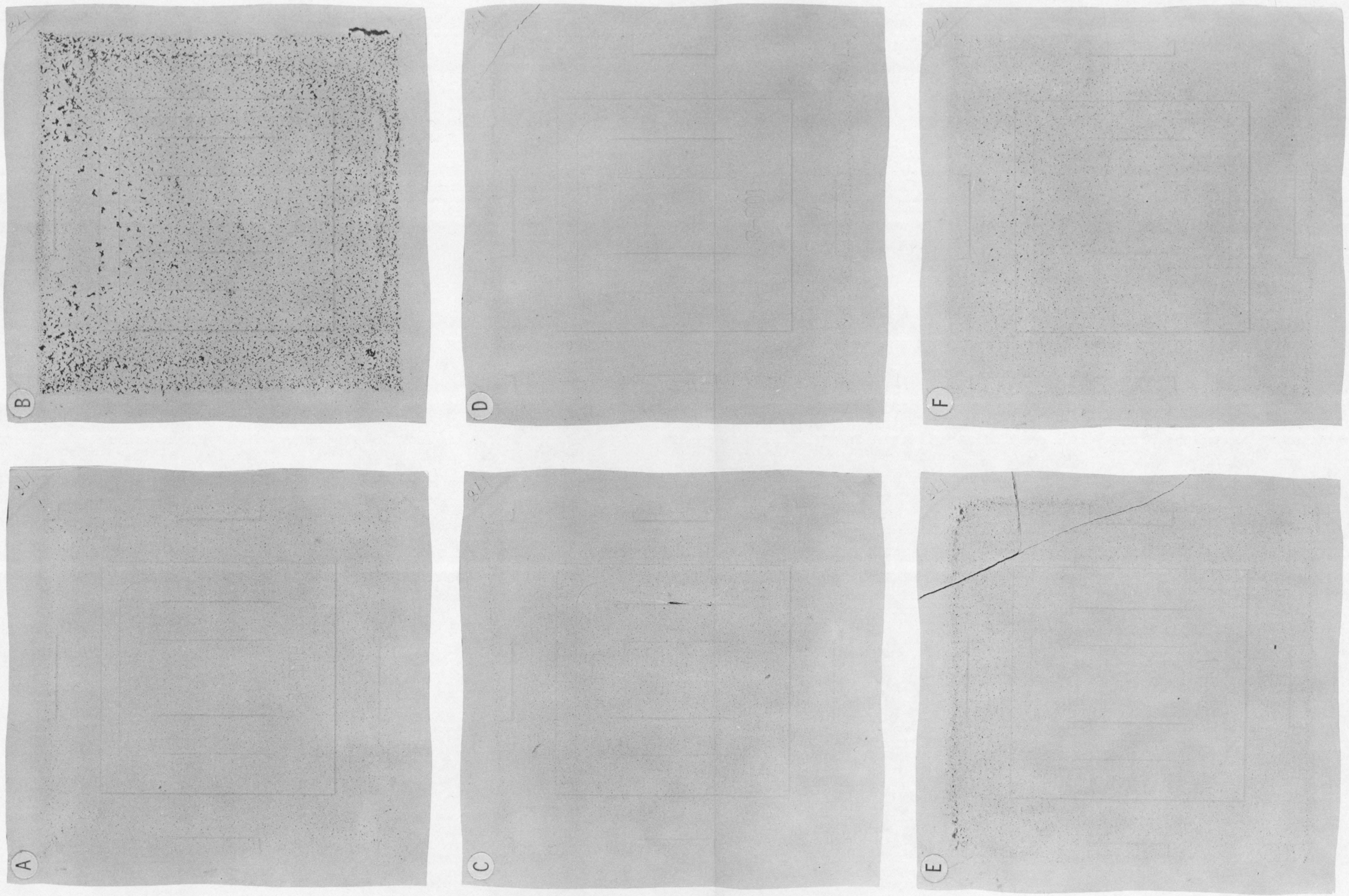

FIGURE 7

Ash Patterns from Bar. No. 178

AEC-GE RICHLAND, WASH.

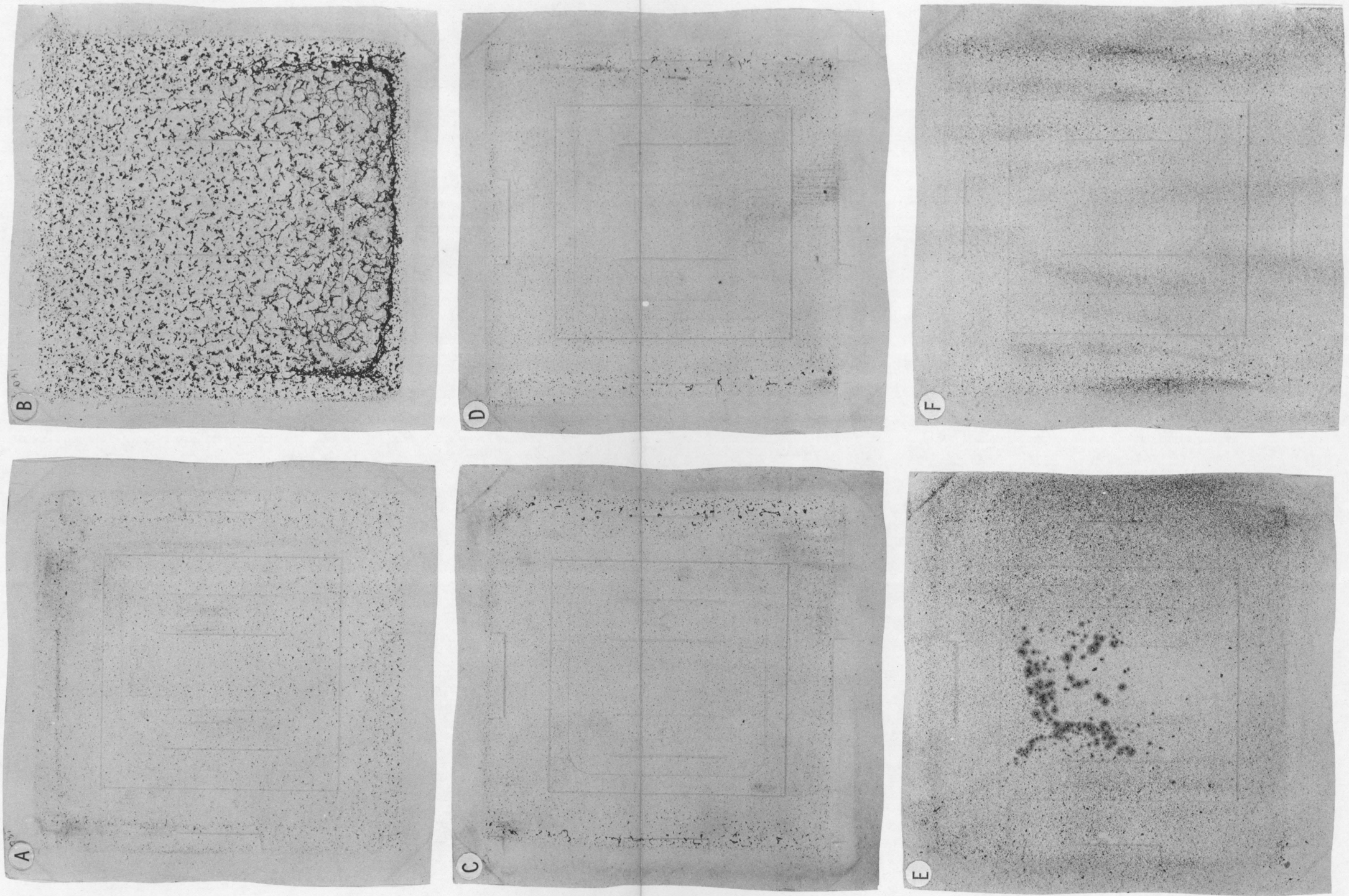

FIGURE 8

Ash Patterns from Bar No. 179

AEC-GE RICHLAND, WASH.

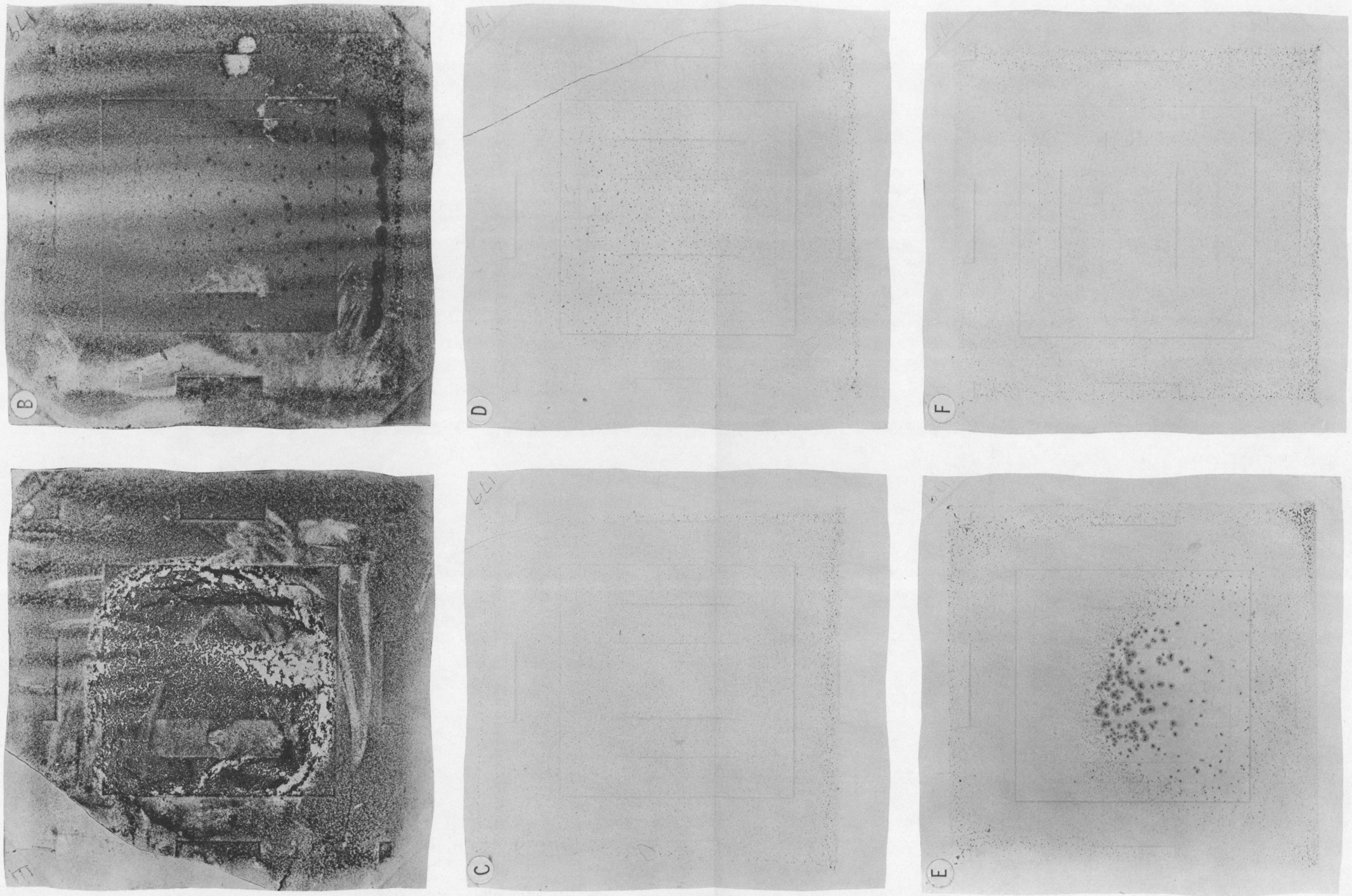

FIGURE 9

Ash Patterns from Bar. No. 180

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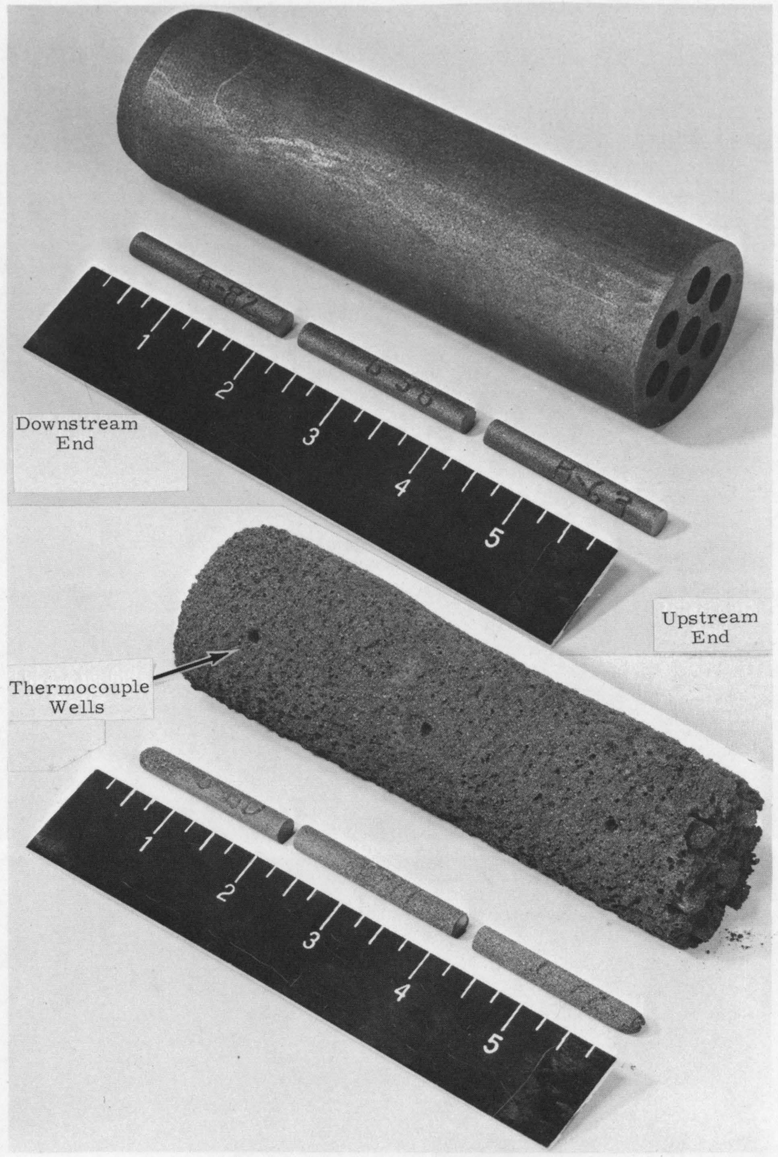

FIGURE 10

Carrier and Samples Used in Determining Relative Oxidation Rates of Various Graphites Shown Before (top photo) and After (bottom photo) Oxidation

Neg. No. 0630350-2, 0630350-1

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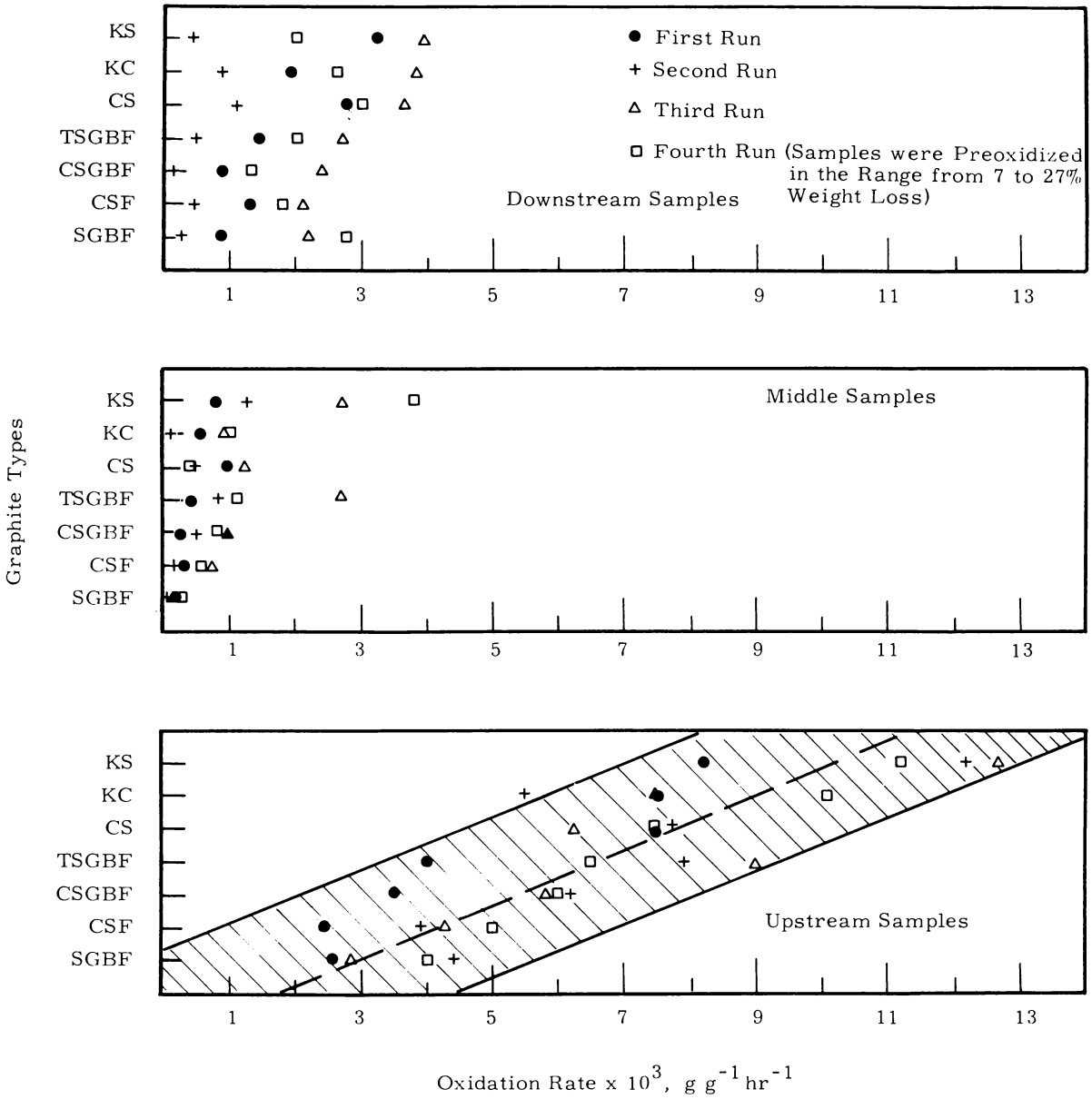

FIGURE 11

Comparison of Oxidation Rates of Various Graphites at 758 ± 4 C in Flowing Air (2 cfh)

AEC-GE RICHLAND, WASH.

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