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PLUTONIUM RELEASE STUDIES

II. Release From Ignited, Bulk Metallic Pieces

By

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TABLE OF CONTENTS

	<u>Page</u>
<u>ABSTRACT</u>	ii
<u>INTRODUCTION</u>	1
<u>PURPOSE</u>	1
<u>SUMMARY</u>	2
<u>EXPERIMENTAL METHODS</u>	2
<u>OBSERVATIONS AND RESULTS</u>	4
<u>Run 1</u>	4
<u>Run 2</u>	7
<u>Run 3</u>	9
<u>Run 4</u>	12
<u>DISCUSSION</u>	13
<u>CONCLUSIONS</u>	15
<u>ACKNOWLEDGEMENTS</u>	15
<u>REFERENCES</u>	16
<u>DISTRIBUTION</u>	17

ABSTRACT

Metallic plutonium pieces ranging in weight from 455.5 to 1770 grams were ignited and allowed to oxidize completely in air with a velocity of 525 cm/sec. Release rates of 0.032 to 0.0045 weight percent per hour were found for the bare metal. Covering the ignited metal during oxidation with magnesium oxide sand reduces the release to 2.9×10^{-4} weight percent per hour. The Median Mass Diameter of the particles airborne during the release from the bare metal was found to be 4.2 microns.

PLUTONIUM RELEASE STUDIES

II. Release From Ignited, Bulk Metallic Pieces

INTRODUCTION

If subjected to the proper conditions of temperature and atmosphere, plutonium metal may ignited and oxidize completely. The fine particulate plutonium oxide formed will be airborne to a greater or lesser degree depending upon oxidation rate, airflow, mechanical working and other variables. When considered in terms of a fire involving plutonium, the degree to which the oxide is released to the atmosphere and the nature of the particles airborne is of great importance in the evaluation of the accident consequences.

A review of available data on the oxidation rate and fractional release of plutonium during overheating was previously reported.^[1] This review also noted the need for further study of the behavior of plutonium and its compounds in overheating situations. The results of the initial experiments using small metallic plutonium pieces has already been reported.^[2] Further experiments are planned to provide additional information useful in the evaluation of the hazards from plutonium and its compounds involved in fires.

In the fall of 1965, the Plutonium Chemistry Section of Isochem, Inc., began evaluating the hazards associated with plutonium metal fires, and suitable extinguishing agents and techniques for use in production and storage areas. During one phase of these studies, large metal pieces were to be ignited and allowed to oxidize completely. This portion of the studies presented a unique opportunity to collect some data on the fine particulate oxide released during the oxidation of these large metallic plutonium pieces. Permission was obtained to collect some limited samples during these runs. This report covers that data obtained.

PURPOSE

The objective of this study was to obtain as much information as possible, under the circumstances, on the amount and nature of the particulate oxide released during the oxidation of large pieces of metallic plutonium at relatively high air velocities.

SUMMARY

Four large metallic plutonium specimens, ranging in weight from 455.5 to 1770 grams, were ignited and ambient air drawn over the ignited metal at ~ 525 cm/sec. The metal was allowed to oxidize completely and the particulate oxide entrained in the air stream collected. Recorded temperatures from thermocouples embedded in the metal ranged from 500°C to 1000°C during the oxidation. From 2.9×10^{-4} to 0.049 w/o of the plutonium was released during the sampling periods (from 22 to 90 minutes) giving release rates ranging from 3.2×10^{-2} to 2.9×10^{-4} w/o per hour. The amount of particulate oxide released in these runs appears to depend upon the phase of the starting metal and the physical conditions during the oxidation.

EXPERIMENTAL METHODS

The collection system utilized in these runs was one which would allow the collection of airborne particles at high air velocities generated by a material at high temperature within the confines of an existing experimental test system. The test system used in the evaluation of plutonium metal fires hazards is described in a memorandum by R. E. Felt and H. D. Merritt.^[3] The test assembly is housed in a free-standing, stainless steel glovebox located in the Plutonium Chemistry Laboratory, 234-5 Building. The hood is 6' long $\times$ 5'9" high $\times$ 26" deep and is divided into two sections (see Figure 1). The first section is divided into an upper and lower area by a stainless steel plate. During these tests, a circular stainless steel cover was bolted over the 22" diameter opening connecting the two areas. The cover was held approximately 1/2" above the floor to permit air to flow from the inlet filter into the burning cubicle. The second section contains a sink which was sealed over during these runs. Other changes were also made to increase the fire protection potential of the facility. The glovebox is shown schematically in Figure 1.

The testing area was partially enclosed by a cubicle mass made of 1/2" transite panels leaving only the front viewing area exposed. The actual oxidation occurred in a 6" diameter $\times$ 1" deep transite ring encircling the ignited metallic piece setting upon an 8" $\times$ 8" $\times$ 1" transite board.

After the metallic piece had been ignited by an arc welding unit, the oxidizing plutonium was placed under the VYCOR[®] chimney and sampling begun. Air was drawn through the chimney by a multistage centrifugal blower at the rate

[®] Registered trade name of the Corning Glass Co.

FIGURE 1
Schematic Diagram of Equipment used in Plutonium Chemistry's Metal Fire Hazards Study

of 40.6 cfm. The system was calibrated prior to its initial use. The VYCOR chimney had an ID of 2.685" (6.82 cm) flaring to 4.5" at the entry. The chimney was 17-3/8" in length with a 9 mm sampling tube used to obtain the electron microscope filter sample ~ 3" from the filter face. The side arm sampler was connected to the chimney by a $\frac{1}{8}$ 24/40 ground glass joint. The flared end of the chimney was positioned vertically to give < 1" clearance between the chimney and transite ring. The system is shown schematically in Figure 2.

Oxide particles airborne were entrained and collected for analysis. At a flow of 40.6 cfm in the 6.82 cm ID chimney, the velocity of the air in the chimney proper was approximately 525 cm/sec (11.7 mph) and had a lifting velocity capable of supporting a spherical particle larger than 200 microns with a density of PuO_2 (11.4). Particles reaching the other end of the filter were captured on a 4" fiber glass filter with a > 99.7% efficiency for 0.3 micron DOP spheres. The particulate oxide was dissolved from the filter using hot nitric acid and the amount of plutonium present determined by alpha counting.

The air flow through the side arm sampler varied from 550 to 800 cm/sec during the sampling period. The particles used for the size distribution analysis were captured on a 13 mm, type AA, Millipore filter. Portions of the filter were placed on electron microscope grids, the filter dissolved with acetone and the particles shadowed. The particles were classified using a Zeiss Particle Size Analyzer.

OBSERVATIONS AND RESULTS

The metal specimens used during these experiments were as-cast ingots or parts of as-cast ingots of alpha or delta phase plutonium. The physical description of all four pieces is listed in Table I. The initial treatment of the metal has been described in the previous section. All four pieces had an 1/8" thermocouple well drilled to the center line prior to use. The response of the metal specimens to the conditions to which it was subjected during the experiment are described below.

Run 1

In this run, a wedge-shaped piece of an as-cast, low density, alpha plutonium ingot was ignited and allowed to oxidize completely. The plutonium specimen weighed 569.8 grams after the thermocouple well had been drilled. The wedge-shaped piece had sides approximately 2" long and was approximately 1" deep. The metal had a density of 17.70 and contained 2000 ppm total metallic impurities.

FIGURE 2
Schematic Diagram Equipment for the Study of Release from Ignited,
Bulk, Metallic Plutonium

TABLE I

PHYSICAL DESCRIPTION OF METAL SPECIMENS

<u>Run No.</u>	<u>Weight (grams)</u>	
1	569.8	A wedge-shaped section of alpha plutonium measuring $\sim 2''$ down a side $\times 2''$ across the curved section $\times 1''$ deep. Metal was low density (17.70) and contained ~ 2000 ppm total metallic impurities. Nominal surface area = 10.5 in^2 .
2	1770	A cylindrical, as-cast, alpha plutonium ingot $\sim 2\text{-}1/4''$ in diameter and $1\text{-}1/2''$ deep. Metal purity was > 99.99 w/o plutonium with < 200 ppm total metallic impurities. Nominal surface area = 29.15 in^2 .
3	997	A hemi-cylindrical, as-cast, delta plutonium ingot with a diameter of approximately $2\text{-}7/16'' \times$ a depth of $1\text{-}5/8''$. Nominal surface area = 21.73 in^2 .
4	455.5	Rough triangular section of an as-cast, alpha plutonium ingot measuring approximately 1 to $1\text{-}1/2''$ down the sides $\times 2\text{-}1/8''$ across the curved section $\times 1\text{-}1/4''$ deep. The metal appears to be stratified and flakes could be pried away with a pair of corceps. Nominal surface area = 12.08 in^2 .

The metal ignited readily and was placed under the chimney during oxidation. The entire upper surface of the piece was ignited by passing an arc-welder slowly over the surface. After the piece was ignited, the specimen was placed under the VYCOR chimney and the sampling begun. Sampling was initiated approximately 5 minutes after the initial heating of the metal. Core temperature at ignition was approximately 375°C. All four specimens appeared to glow brighter after air flow was initiated. The metal temperature increased rapidly to a maximum (960°C), dropped off and rose again. The temperature profile is shown in Figure 3.

Initially the metal specimen retained its original shape with occasional, small chunks of oxide falling away from the main body of material. Approximately 10 minutes after ignition, the specimen began to lose its shape and some airborne sparks were observed. Oxidation above ignition temperature appeared to be complete within 40 - 60 minutes after ignition.

Sampling was continued for approximately 30 minutes. After the oxide mass returned to near ambient temperature (50°C - 60°C), the filter was removed for analysis. The filter had a moderate amount of yellowish-brown powder over its entire sampling area with an occasional black speck. The amount of plutonium collected during the 90 minute sampling period was found to be 0.268 grams, 0.049 weight percent of the starting material. This indicates an average rate of release during ignition and cooling of 3.2×10^{-2} weight percent per hour.

Run 2

In Run 2 a cylindrical, as-cast, high purity alpha plutonium ingot was ignited and allowed to oxidize completely. This was the largest metal specimen weighing 1770 grams. The ingot had a diameter of approximately 2-1/4" and was about 1-1/2" deep. The metal was greater than 99.99% plutonium with less than 200 ppm total metallic impurities.

Some difficulties were encountered in igniting the piece and a longer heating period was required probably due to the mass of metal involved. Initially, the entire top surface of the ingot was ignited. Within two minutes the entire mass collapsed and molten, incandescent, plutonium flowed over the transite plate. Sampling was begun immediately after ignition and samples for size distribution begun after 5 minutes. Three separate samples were taken for size distribution analysis to provide a range of particle population for use in the electron microscope -- 1, 5 and 10 minutes. The 10 minute sample was used for analysis. Some airborne sparks were observed during sampling.

FIGURE 3
Time vs Temperature for Plutonium Oxidation Runs Above Ignition Temperature on Large Metal Pieces

The measured temperature following ignition of the specimen exhibited a profile similar in shape to that of Run 1, (see Figure 3). A core temperature at ignition of 510°C was recorded rising rapidly to approximately 950°C. A maximum of 1000°C was recorded during oxidation above ignition temperature. Again the intensity of glow increased after airflow over the sample was begun. Oxidation above ignition appeared to be complete in approximately 40 minutes. The temperature during the cooling period decreased at a much lower rate than in the previous run, probably due to the mass of oxide involved.

The plutonium airborne was sampled for 45 minutes almost wholly during the oxidation above ignition temperature. After cooling, the filter was removed and contained a moderate amount of yellowish-brown powder over the entire sampling area. Upon analysis, the filter was found to have 0.257 grams plutonium representing 0.014 weight percent of the starting metal mass. This gives a release rate of 0.019 w/o per hour.

The 10 minute sample taken via the side arm was analyzed by electron microscopy. The projected diameters of 650 particles were determined using a Zeiss Particle Size Analyzer. The number of particles less than a stated size was plotted against projected diameter on log probability paper, and a Count Median Diameter (CMD) of 1.2 microns determined. This plot is shown in Figure 4. Using a computer program devised to convert CMD into MMD (Median Mass Diameter) for log-normally distributed, a MMD of 686 micron was calculated. It is apparent from Figure 4 that the particles are not log-normally distributed and the geometric standard deviation (σ) of 4.3 does not represent the slope of the size distribution.

The MMD was recalculated by assuming the material is homogenous with a density of 11.4 and the volume is a cube function of the projected diameter. The replotted data is shown in Figure 5 and gives a MMD of 4.2 micron. The shape of the size distribution curve is similar to those found in previous experiments using metallic specimens^[2] and indicates the presence of more than one size distribution.

Run 3

Run 3 was the ignition of a hemi-cylindrical, as-cast, delta plutonium ingot weighing 997 grams. The metal specimen had a diameter of 2-7/16" × 1-5/8" deep. In this experiment, the turnings generated during the drilling of the thermocouple well were added to the metal mass and used during the run.

FIGURE 4
Particle Size Distribution by Number of Oxide Entrained in Run 2

FIGURE 5
Particle Size Distribution by Weight of Oxide Entrained in Run 2

The metal appeared to ignite readily and the entire top surface of the piece was glowing within two minutes by passing the arc welding torch slowly over the surface. During this period, the turnings ignited and oxidized completely although they were not directly in the arc welder flame. Within a few minutes of ignition, the oxide coat of the metal specimen cracked and molten plutonium flowed to the transite floor. The temperature profile during ignition and oxidation above ignition temperatures was similar to the previous two runs (see Figure 3). There was an initial rapid rise in temperature, a decrease and a higher level which was maintained until the end of the oxidation above ignition. Maximum core temperature recorded during the run was 930°C.

Sampling was initiated approximately four minutes after heating of the sample was begun. Particles airborne were collected for 22 minutes and the entire sampling period was during oxidation above ignition temperature. The sampling period did not include the rupture of the oxide coat nor the oxidation of the turnings. After the oxide mass returned to near ambient temperatures, the filter was removed for analysis. The filter contained a moderate amount of dark (black or olive green) powder. Analysis indicated the presence of 0.034 grams of plutonium which is 0.0034 weight percent of the original weight of metal. This yields a release rate of 0.0045 w/o per hour.

Run 4

Run 4 was the ignition and oxidation of a triangular base prism of an as-cast low quality, alpha plutonium ingot. Soon after ignition (approximately 5 minutes) the ignited specimen was covered with magnesium oxide sand to smother the fire; a procedure successful in previous tests. The metal specimen measured approximately 2-1/8" x 1-1/2" x 1" on its three sides and was 1-1/4" deep. The weight of the specimen was 455.5 grams. The metal was stratified and layers were plainly visible. Flakes of metal could be pried free of the main mass by insertion of a pair of forceps between the layers. No analysis of the actual quality of the metal was available.

The initial temperature behavior of the metal was similar to all previous runs. The metal ignited readily -- within 1 minute. Soon after ignition, the oxide coat cracked and molten plutonium flowed onto the transite floor. This material did not remain ignited. Core temperature was approximately 520°C at ignition. The glowing metal was covered with MgO sand and the entire mound of metal and sand placed immediately under the VYCOR chimney and air sampling begun. There was an initial rapid drop in temperature from 800°C to 570°C in

one minute and a low of 495°C in three minutes. After this low, the temperature rose to 635°C and showed a steady decrease not exhibiting the rapid drop in temperature after oxidation shown by the three previous runs. The maximum core temperature during this portion was 650°C.

Sampling of the fine particulate oxide airborne was begun approximately 4 minutes after ignition occurred and soon after the specimen was covered with MgO sand. The sampling period was 60 minutes. Upon removal from the holder, the particle filter had a fine grey film over its sampling surface. The weight of the plutonium deposited was found to be 0.00132 grams constituting 2.9×10^{-4} w/o of the starting metal. The release rate is also 2.9×10^{-4} w/o per hour.

After the completion of Run 4, a control filter was manipulated through the procedure followed with the actual runs to determine the amount of contamination pick-up which could occur. The control filter contained 0.00054 grams of plutonium representing 0.2, 0.2, 1.5 and 41 percent of the weight of plutonium found on the four filter samples.

TABLE II

PLUTONIUM RELEASED DURING THE OXIDATION OF LARGE
METAL PIECES ABOVE IGNITION TEMPERATURES

<u>Run No.</u>	<u>Weight Plutonium (grams)</u>	<u>Weight Pu Released (grams)</u>	<u>Weight % Plutonium Released</u>	<u>Total Time Sampled (min)</u>	<u>Release Rate (w/o hr)</u>
1	569.8	0.268	4.9×10^{-2}	90	3.2×10^{-2}
2	1770	0.257	1.4×10^{-2}	45	1.9×10^{-2}
3	997	0.034	3.4×10^{-3}	22	4.5×10^{-3}
4	455.5	0.00132	3.9×10^{-4}	60	2.9×10^{-4}

DISCUSSION

The four plutonium specimens used in these experiments ignited and oxidized similarly in several respects. All four show temperature "spikes" reported by Wanklyn^[4] and Hilliard^[5] for the ignition of metallic uranium and by Schnizlein and Fisher^[6] for the ignition behavior of plutonium and some of its alloys. The initial heating period was required to heat the entire mass of metal to the ignition point. Once ignited, the heat generated by the oxidation increased

the temperature until radiative and convective heat losses either hold it at that temperature or cause the temperature to fall. In Runs 1 and 2 in which alpha plutonium was used the maximum temperature was approximately 1000°C. In Run 3 in which delta plutonium was used, the maximum temperature was 930°C. The 40°C difference between Runs 1 and 2 probably reflects the differences in mass between the two metal specimens. The lower temperature found in Run 4 shows the effect of a large mass of cool material upon the temperature during oxidation. The effects of the magnesium oxide appear to be two-fold -- to draw heat away from the metal and limit the air flow to the metal. If either effect is sufficient, the metal cools and ignition does not occur. Under the condition of this test, the air flow past the MgO-covered metal must have been sufficient to diffuse enough air down to the metal to maintain ignition. The maximum temperature possible under these conditions was 350°C lower than in Runs 1 and 2 in which similar material was used under the same conditions. This lower maximum temperature probably reflects the slower oxidation rate, resulting from lowered air supply due to interference from the MgO sand.

When the heat produced by the oxidation was not sufficient to maintain the ignited condition, the temperature dropped rapidly. This is shown by the behavior of Runs 1 through 3 in Figure 3. The tailing shown probably reflects the surface area, heat transfer characteristics and the mass of the oxide produced. The picture of Run 4 is complicated by the presence of the MgO and the end of ignition is not as obvious as the first 3 runs.

The release of fine particulate oxide under these conditions appears to be affected by the phase of the starting metal and physical surroundings during oxidation. Run 1 and 2 are both alpha plutonium. The release from Run 1 is approximately twice that found in Run 2. The weight of the starting material used in Run 2 is three times that used in Run 1 and the difference may also reflect a difference of area of oxide exposed or may indicate a higher release rate during the cooling period after ignition. The release from Run 3 using delta plutonium is 1/5 to 1/10 that of either Run 1 and 2, indicating the release from delta metal is lower than from alpha metal under these conditions. The release from Run 4 is less than 1/10 that of any other run and shows that MgO sand is an effective barrier for the particles produced. The amounts released in all four runs are well within the "conservative" estimate of approximately 0.1 w/o given by Stewart^[7] for the release from metal on ignition with oxidation above the melting point in a dynamic system. Also the air velocity may be high for fire situations indoors but is less than the velocities

found outdoors on many occasions. Using a calculated nominal surface area, the release rates found for these four runs did not indicate a surface area dependency.

The sizes of the particles airborne under these conditions is surprisingly small. The air velocity used (525 cm/sec) is theoretically capable of supporting a particle of greater than 200 micron diameter of the density of PuO_2 . The largest particle found on the electron microscope was approximately 8 microns in diameter. Undoubtedly some particles between 8 and 200 microns in diameter were formed. Both Mishima^[2] and Stewart^[7] have reported their presence in the residue from the air oxidation of metallic plutonium. Either the particles of this size range are present in the mass of oxide and trapped by the surface or the particles reported previously are formed during some subsequent period.

The physical arrangement and dimensions of the collecting chimney and the flow rate were selected with the intention of providing a velocity high enough over the specimen and in the chimney to entrain and carry away quite large oxide particles. The velocity of about 12 miles per hour in the chimney and of the order of half this across and around the specimen are believed to be realistic air flows which might be experienced in accidents involved with the overheating of plutonium. It was not possible in this brief study conducted as an adjunct to a separate study to investigate the parameters of flow rate on total release. The reported observations must be interpreted as being indicative rather than conclusive.

CONCLUSIONS

The amount of fine particulate oxide airborne during the oxidation of metallic plutonium above ignition temperatures in air at a velocity of 525 cm/sec ranges from 0.032 to 0.0045 weight percent per hour. In these experiments, the amount released by delta stabilized metal was lower than from the alpha phase metal. The MMD of the particles airborne was 4.2 micron with no particles greater than 8 microns detected.

Immersing the ignited metal in magnesium oxide sand can reduce the amount released by an order of magnitude.

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