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LOS ALAMOS SCIENTIFIC LABORATORY
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by

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ABSTRACT

Ternary eutectic fluoride and graphite fines were used on plutonium fires to determine their relative effectiveness as plutonium fire extinguishants. These tests confirm this Laboratory's use of graphite fines for extinguishing small fires. For larger (more than 200 grams) or more scattered fires, ternary eutectic fluoride extinguishant might offer some advantage over the graphite fines because of its tendency to form an adherent. noncollapsing layer.

Fires involving even small amounts of plutonium can cause major losses through airborne contamination of work areas which necessitates extensive decontamination and operational downtime. The control and extinguishment of these fires is of paramount importance to any laboratory using plutonium.

Plutonium, a high density, radioactive, pyrophoric metal, is always processed or worked in completely encased glove enclosures, commonly called glove boxes (Fig. 1). These glove boxes can be inerted with argon or helium, and, if the box contains over 50% inert gas (<10% O_2), there is little danger of the plutonium turnings igniting during machining. However, since ignition of plutonium turnings or fine pieces is possible and could burn the enclosure gloves, thus releasing serious contamination. it is necessary to quench these fires.

At the Los Alamos Scientific Laboratory (LASL), graphite fines produced in machining of high purity graphite have been the extinguishing agent of choice for plutonium and uranium fires. A commercial graphite agent is not used because it contains impurities that complicate the recovery of these expensive metals.

For some time, the British have been impressed with the capabilities of a powdered ternary eutectic chloride and a ternary eutectic fluoride for extinguishing metal fires. Of the two, the fluoride is the more effective on plutonium.* In view of the favorable British reports, LASL conducted tests of the ternary eutectic fluoride to compare the effectiveness of the eutectic and of our graphite fines as extinguishing agents.

The composition of the ternary eutectic fluoride (TEF) powder is:

46.5 mole % lithium fluoride
11.5 mole % sodium fluoride
42.0 mole % potassium fluoride

This material as received was a white, hygroscopic powder. At present, John Kerr and Company, Ltd.. of Liverpool. England, appears to be the only source. They seal TEF powder in plastic bags in 1- and 2-½ lb. lots. The current cost is $2.80 to $4.20 per pound. FOB, Liverpool.

Fire extinguishing tests were planned with the LASL Plutonium Fabrication Group. The following conditions were applied.

1. Fires involving unalloyed plutonium and an alloy containing 57.7% plutonium,

*J. Holliday and W. A. Conway, "The Extinguishing of Plutonium Fires," TRG Report 342(D), United Kingdom Atomic Energy Authority. Risley. Warrington, Lancashire, England (1962).

9.4% cobalt, and 32.9% cerium were used to test the TEF powder and graphite extinguishants. The total weight of metal used per test was limited to 250 grams. The Pu-Co-Ce alloy (a proposed reactor fuel) was in the form of small chunks and pieces of 0.3-in.-diam rods; the plutonium was one ⅜-in.-diam rod, and fresh 0.005- to 0.008-in.-thick turnings, not wider than 5/32 in.

2. Temperature vs time measurements were taken. The extinguishants were applied when all the metal was seen to be burning, not at any preselected temperature.

3. Tests were conducted in a glove box. Glove box atmosphere was dry air maintained at 0.5-in. water negative pressure. The moisture content of the glove box atmosphere was less than 50 ppm H_2O.

4. The metal was ignited by heating it in a thermocouple-equipped tray of 0.1-in.-thick stainless steel (4-½ × 5-¼ × ½ in. deep), on a 1200-watt hot plate.

5. Two Chromel-Alumel thermocouples were attached to the burning tray. One was encased in magnesium oxide insulation, the other in a stainless steel sheath. (Since the magnesium oxide insulated thermocouple did not respond so fast to temperature changes, only data from the other thermocouple are reported here.)

Screen sizes of powder (percent extinguishant retained on screen):

Screen Size	TEF	Graphite
10	0	0.46
20	0.05	68.63
50	80.26	9.44
80	8.32	4.66
170	7.16	7.74
200	0.79	0.73
400	2.14	4.87
pan	1.28	3.47

In the first test (Figs. 2 and 3), 140 grams of the Pu-Co-Ce alloy was ignited. The melting and ignition temperatures of this alloy are quite close, about 450°C. When the metal appeared to be burning uniformly, TEF powder was sprinkled onto the fire manually from a large metal salt shaker. Initial application of the TEF powder caused a mild flame to appear, but it quickly subsided. The recorded metal temperature was 473°C when the TEF powder was applied. The temperature decrease was recorded until the fire was observed to be extinguished. Thirty-one grams of TEF powder was applied. The quench rate (metal temperature drop per elapsed time in seconds) was calculated to be about 2°C/second. Some crusting of the TEF powder was observed.

The second test (Figs. 4 and 5) involved the plutonium rod and turnings. The turnings ignited, but the rod did not. The recorded temperatures were low, perhaps because the rod may have acted as a heat sink. Initially 16 grams of turnings and the 91-gram rod were used. Later, 52 grams of fresh turnings were added so that the test could be continued. When the turnings were burning, TEF powder was sprinkled on them. The recorded metal temperature was 440°C when the TEF powder was applied. Forty-eight grams of TEF powder was used. A quench rate of 1°C/second was established. No crusting of the TEF powder was observed.

The third test involved 80 grams of plutonium turnings. TEF powder was dumped on the fire in one motion when all the turnings appeared to be burning. At this time the recorded temperature was 645°C. No attempt was made to determine the minimum amount of TEF powder required to extinguish the fire. One hundred and fifty-one grams was applied. A quench rate of 6°C/second was established. A fragile crust of TEF was formed on the turnings.

The fourth test (Figs. 6 and 7) involved 80 grams of plutonium turnings. The extinguishing agent was graphite fines. The graphite was applied when the recorded metal temperature reached 603°C. Four hundred and seventy grams of graphite was dumped into the fire in one motion. A quench rate of 4°C/second was established. The graphite did not form a crust over the burning metal.

The following observations were noted:

1. Disturbing the layer of extinguishant to expose the hot metal may result in reignition.

2. Crusted TEF does not collapse when the metal burns from under it. Graphite, on the other hand, does collapse, and small sink holes were visible on the surface.

3. Whereas light application of TEF powder resulted in a short-lived, soft flame, fast application did not. A wispy white smoke

was generated and continued until the fire was out. The smoke, however, did not appear to be corrosive and did not harm the interior of the glove box.

4. When TEF powder was used to extinguish the plutonium turnings, practically all the metal was reduced to oxide. When graphite was used, however, dark turnings were very much in evidence and much less oxide was formed.

5. The 1-lb and 2-½-lb plastic containers of TEF powder are designed to be applied to a fire as units. For the type of fire we envision at LASL, this application involves much more TEF than necessary. Also, TEF powder is hygroscopic and may require storage in some type of air-tight container.

6. It is apparent that the application of either TEF powder or graphite fines to a plutonium fire will prevent a burn-through. Our experience is that burning plutonium in contact with stainless steel (a glove box floor) forms a eutectic having a melting point well below that of steel or plutonium.

7. Despite the lower recorded temperatures from the thermocouple, the actual surface temperatures of the burning metals exceeded 700°C as estimated from pyrometric experience.

	Test Number			
	1	2	3	4
Metal	Pu-Co-CE	Pu	Pu	Pu
Metal Wt. (g)	140	159[a]	80	80
Metal Temperature at Start of Test (°C)	473	440	645	603
Metal Temperature at End of Test (°C)	225	285	238	288
Time to Extinguish (sec)	106	150	72	72
Extinguishant	TEF	TEF	TEF	Graphite
Wt. of Extinguishant (g)	31	48	151	470
Quench Rate (°C decrease in metal temp.) per Second	2	1	6	4
Application of Extinguishant	sprinkle	sprinkle	pour	pour
Crust Formed	some	none	some	none

[a]Explained in text; consider that 52+ grams was actually burning.

SUMMARY

Ternary eutectic fluoride powder was found to be effective as an extinguishant for small plutonium fires. However, the powder is expensive, and, because it is hygroscopic, may have to be kept in air-tight containers. Graphite fines were also found to be effective in extinguishing such small fires, and have the advantages, at least as far as LASL is concerned, of being essentially free of cost and of storage problems. It is possible that TEF powder, because of its high heat of fusion and tendency to form an adherent, noncollapsing layer on the burning metal, might offer some advantages over the graphite fines in controlling fires scattered over a wide area or involving more than 200 grams of plutonium. In general, these test results reaffirm our confidence in the use of graphite fines in controlling the usual type of plutonium fire, about 200-gram quantities, encountered at LASL. The use of either graphite fines or TEF powder would not complicate the recovery of plutonium.

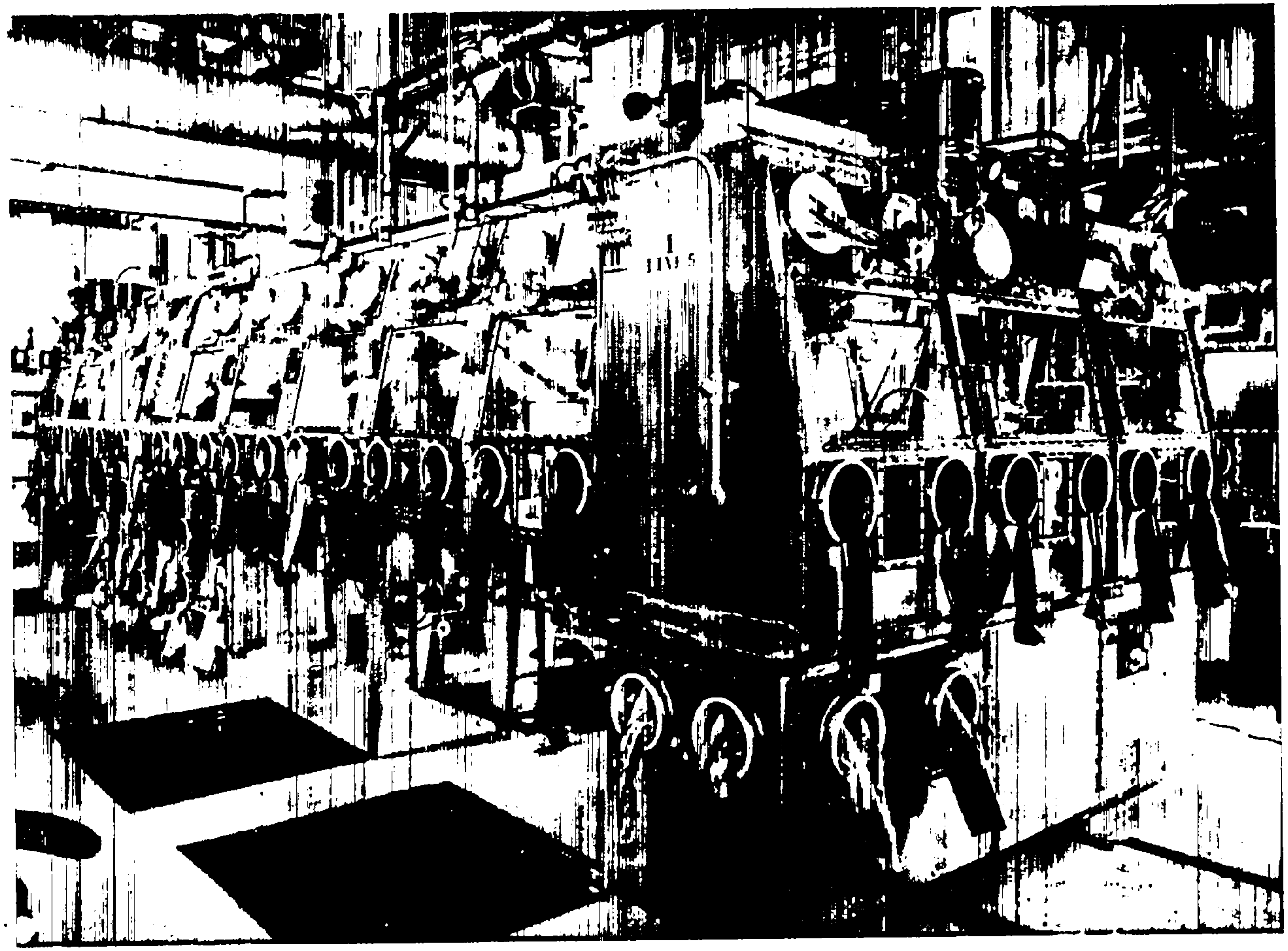

Fig. 1. Glove boxes for processing and working plutonium.

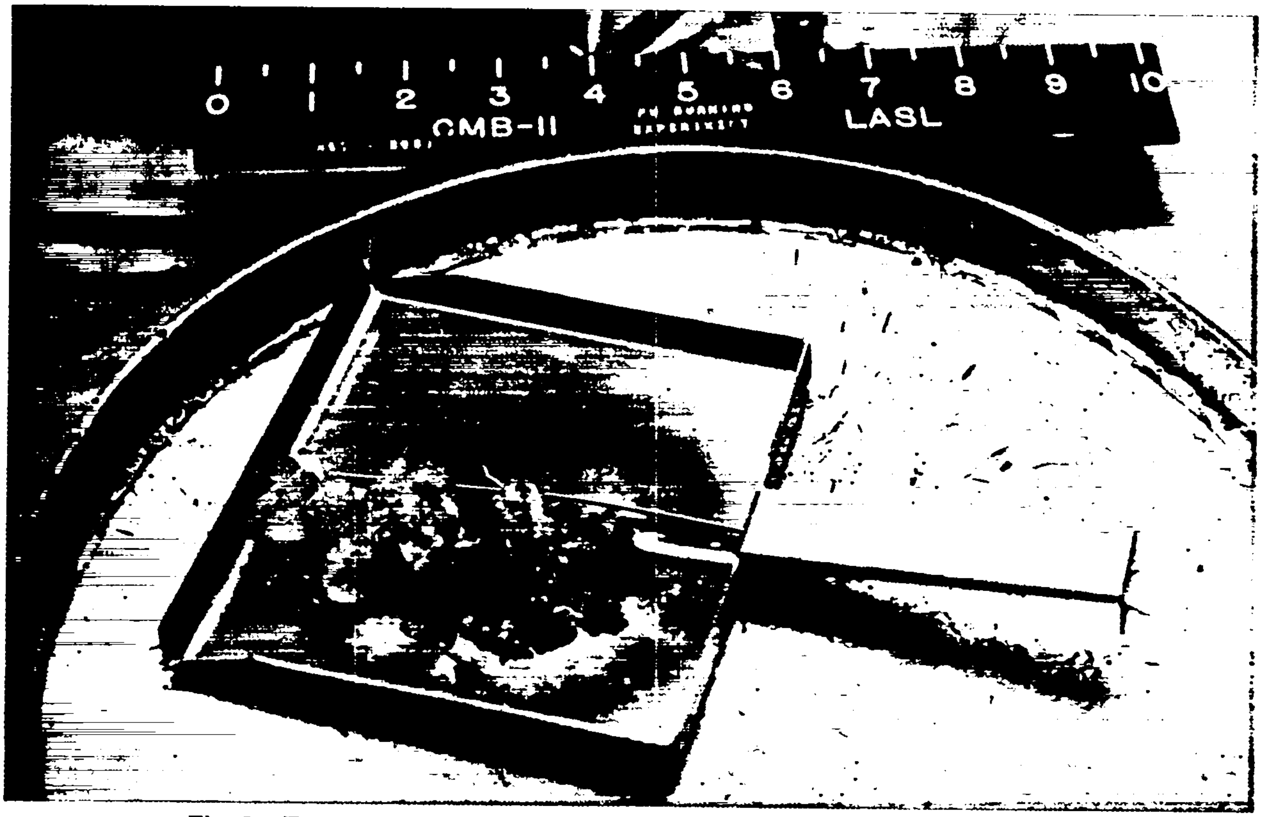

Fig. 2. Test No. 1: Burning Pu-Co-Ce alloy just before TEF powder application.

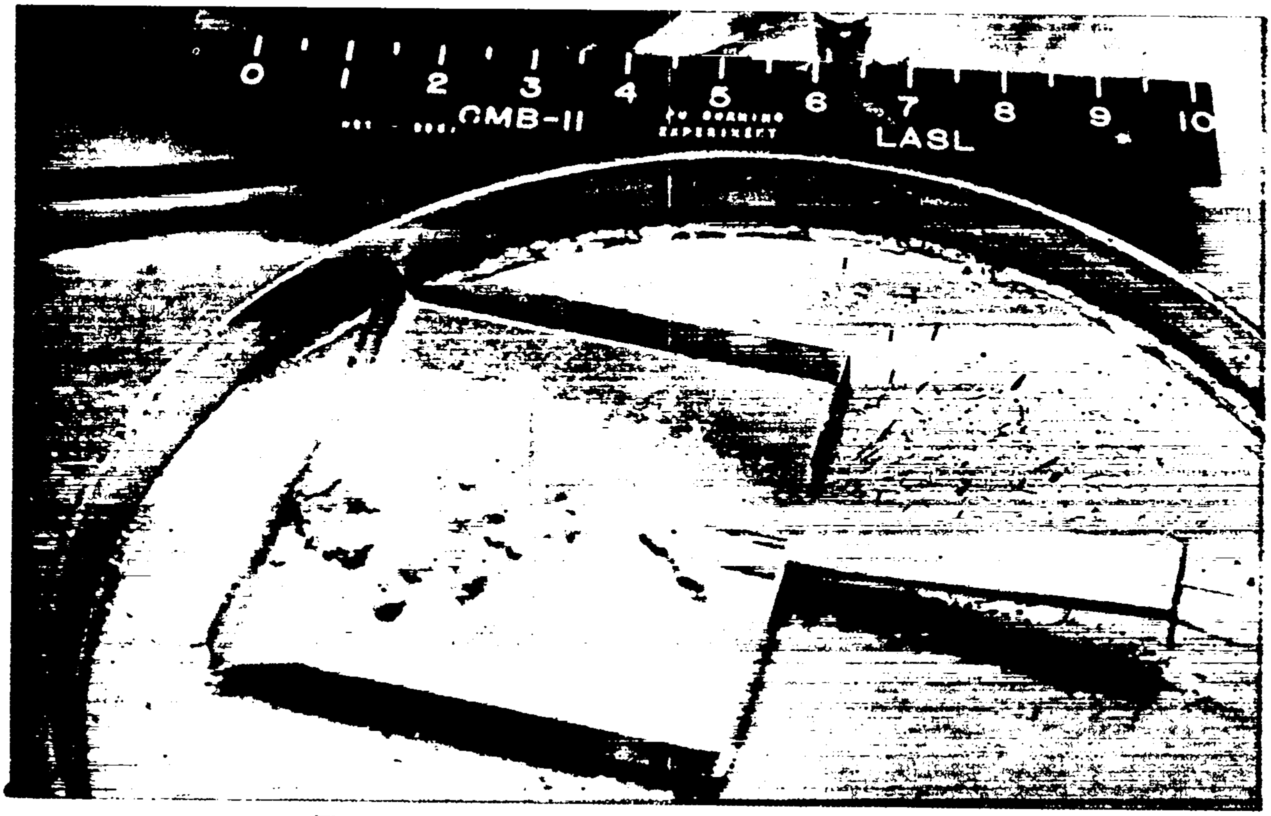

Fig. 3. Test No. 1: TEF powder applied to Pu·Co·Ce fire.

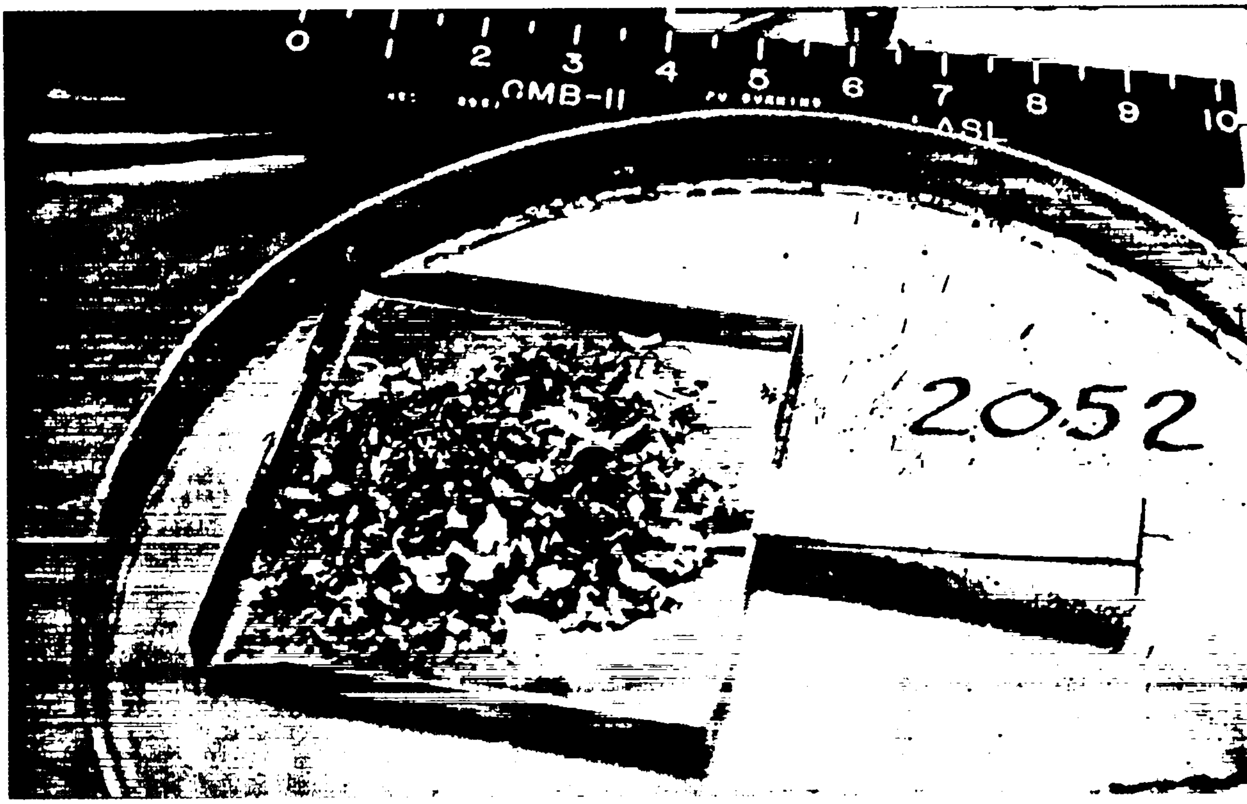

Fig. 4. Test No. 2: Burning plutonium just before TEF powder application.

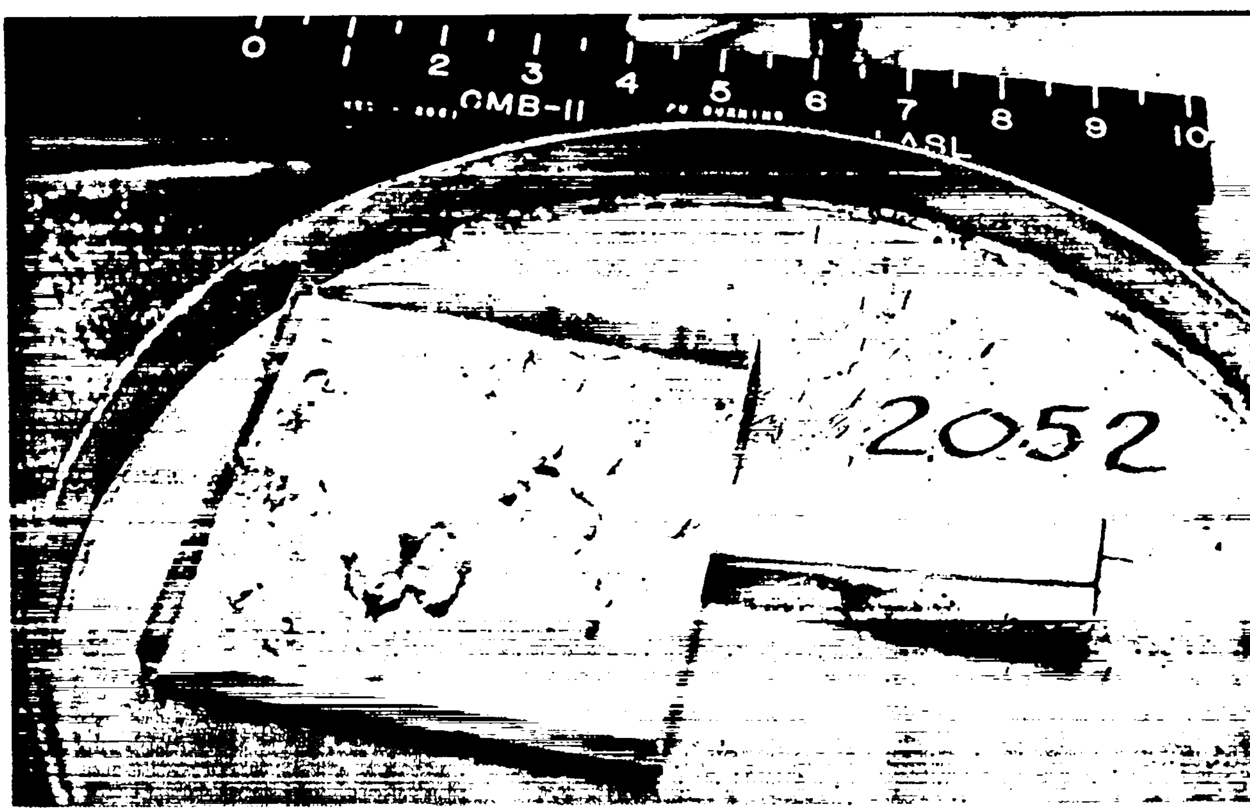

Fig. 5. Test No. 2: TEF powder applied to plutonium fire.

Fig. 6. Test No. 4: Burning plutonium turnings just before graphite fines application.

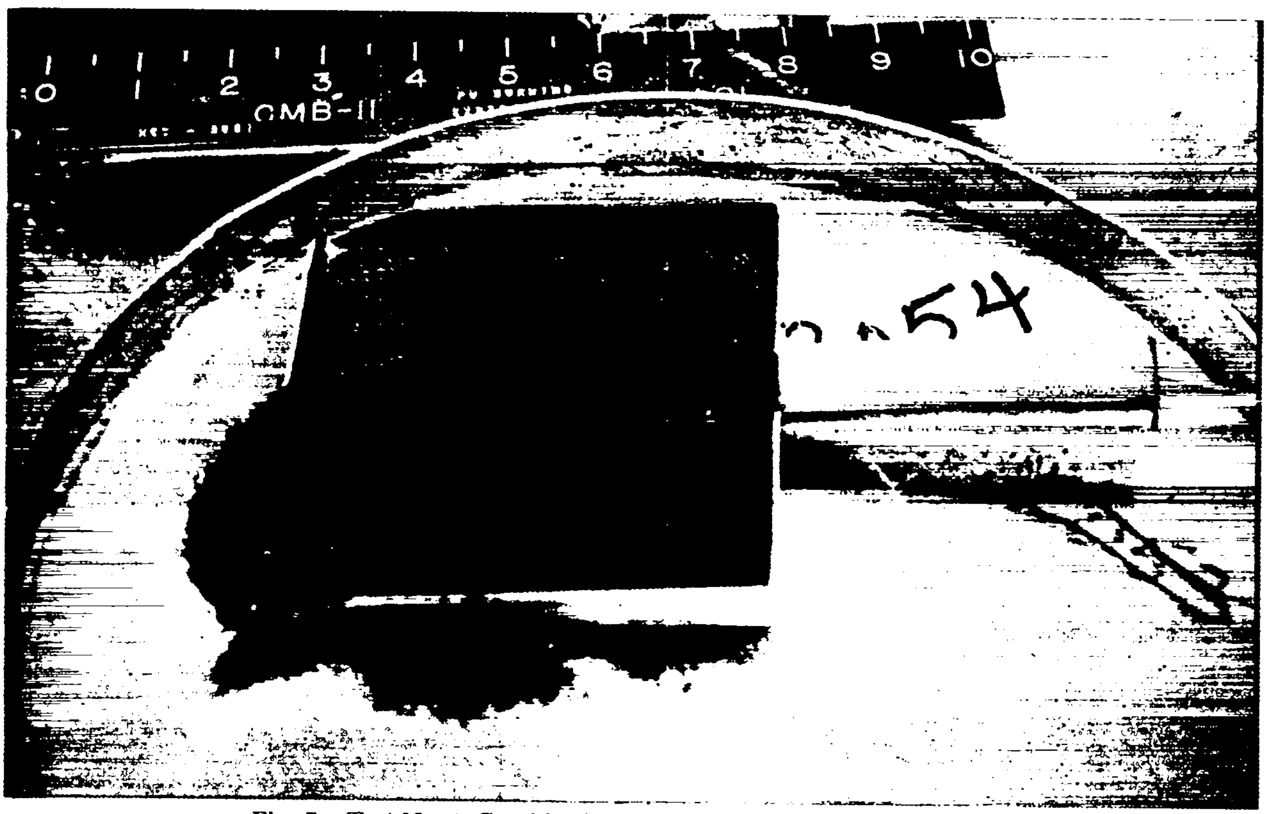

Fig. 7. Test No. 4: Graphite fines applied to plutonium turnings fire.