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SEP-171

PRELIMINARY REPORT ON THE PREPARATION
AND USE OF NON-PYROPHORIC URANIUM POWDER

By
R. B. Wagner
A. Bartoszak
S. Storchheim

August 9, 1954

Atomic Energy Division
Sylvania Electric Products, Inc.
Bayside New York

Technical Information Service, Oak Ridge, Tennessee

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METALLURGY AND CERAMICS

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Metallurgy & Ceramics

ABSTRACT

Preliminary results have shown that the pyrophoric nature of uranium powder can be inhibited by additions of either paraffin wax or paraffin oil. The resultant mixtures can be safely handled in air and can be processed in air into fuel elements of acceptable density and chemical composition.

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I. INTRODUCTION

The development of non-pyrophoric uranium powder would considerable simplify the design and operation of equipment used to fabricate powder into finished forms. The cost of producing powder metallurgy fuels would thus be materially reduced.

There has been definite evidence that inhibition of the pyrophoric nature of uranium powder is possible⁽¹⁾. In addition, cursory studies at Sylvania have indicated that certain reagents will inhibit the pyrophoricity of fine uranium powder. This type of powder, after inhibition, can then be handled in air and still produce slugs of acceptable densities, 18.86 g/cc and better.

The following preliminary report contains the results of a more detailed investigation concerning the inhibition of the pyrophoric nature of uranium powder. In addition, details concerning the further processing of these resultant mixtures are described.

II. MATERIALS AND PROCEDURES

A. Materials

All experimental work of this investigation was conducted on the following types of uranium powder produced by a hydriding process.

1. -12 mesh derby powder.
2. -80 mesh ingot powder.
3. Ingot powder of various mesh fractions.

The untreated powder was, at all times, stored under an argon atmosphere in closed containers. The powder was processed as soon as possible after production. The time elapsed between the production of the powder and its combination with the paraffin did not exceed several days.

The paraffin wax used in the experiments was a U.S.P. grade, medium; the paraffin oil used was a U.S.P. grade white medium heavy.

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B. Procedures

1. Application of Paraffin Wax or Oil to Powder

a. Tumbling Method

It was attempted to coat the uranium powder particles with a protective coating of paraffin wax by tumbling the powder with wax shavings. Powder of various mesh fractions was tumbled in charges of 20 to 500g, under a protective atmosphere of tank argon, with 1-1/2 to 5% of paraffin wax shavings in sealed glass jars on a standard tumbling mill. The tumbling was carried on for varying lengths of time from 1-1/2 to 24 hrs., and at temperatures near to and above the melting point of the wax, as well as at room temperature.

b. Solute Deposition Method

A phase of the investigation consisted of the surface coating of uranium powder with paraffin wax by a "wet" or "solution" method. The solution consisted of an inert solvent, such as kerosene or benzene, which contained from 1 to 10% (by weight) dissolved paraffin wax. The powder was wetted by pouring it, under a protective atmosphere of argon, into the solution. The solvent was subsequently removed by evaporation in air or under vacuum, leaving the wax solute as a surface coating on the powder.

c. Slurry Method

The major phase of the investigation centered on the protection of the pyrophoric uranium powder by submersion in molten paraffin wax (with or without subsequent solidification) or paraffin oil during processing. The procedure for obtaining the slurry consisted simply in pouring the dry powder into the liquid paraffin under a protective atmosphere of argon. Care was exercised to assure the complete wetting of the powder by the paraffin. This is best accomplished by agitation during the mixing of the components. The resulting slurry was subsequently processed in air.

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2. Evaluation of Pyrophoric Properties of Paraffin-Treated Powder

The following simple apparatus was used for the determination of the ignition temperature of the paraffin-treated powder. The apparatus consisted of a 1/2" thick graphite slab heated by an electric hot plate. A thermocouple, connected to a potentiometer, was placed on the slab at its center. The treated powder was poured through a funnel from a standard height on the thermocouple junction. The resultant uniform cone of powder was heated by conduction from the heated graphite slab. The point at which the powder started to ignite was taken as the temperature of spontaneous ignition of the powder. These tests were performed under a hood and the operators' safety was insured by the use of protective shielding and asbestos clothing. Powder samples not treated with wax were processed in conjunction with the treated samples and checked for spontaneous ignitability at room temperature. The latter test served as a control.

3. Pressing of Standard Slugs

The paraffin-treated powder, when found not to be pyrophoric at room temperature, was subsequently fabricated by hot pressing in air or in vacuum, into slugs 4" long by 1.440" in diameter. The hot pressing was, in the case of the tumbled powder, performed directly after treatment or subsequent storage without intermediary processing. In the case of the slurry type of powder, preforms of the same shape, but slightly smaller diameter (1.370") than the hot-pressed slugs, were cold-pressed to remove the greater part of the paraffin wax or oil. These preforms were subsequently hot-pressed according to a standard technique. The techniques and procedures used in these operations can be summarized as follows:

a. Preforming

Standard, 16" long inconel hot-pressing dies, with split insert, were used to compact 1700 to 1800 gm of powder to dimensions approximately 1.37" in diameter by 5-1/2 - 7" long, varying in accordance with the applied loads. The die, cold in the case of a paraffin oil slurry, warm in the case of a paraffin wax slurry, was loaded in air and pressed, single or double-action, at various pressures. Careful oxidation and lubrication of the die wall and punches with Aquadag was necessary to insure smooth, uniform pressing.

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b. Hot-Pressing of Powder or Preforms

(1) In Vacuum

The standard hot-pressing equipment and technique for hot-pressing uranium slugs was utilized to fabricate compacts from treated powder. Untreated powder was hot pressed for control purposes. All compacts were pressed at 12 tsi for 15 minutes at 600°C in vacuum.

(2) In Air

In practically all instances of hot-pressing slurry preforms, the hot-pressing operation was performed in accordance with standard procedure, but without the use of a vacuum; i.e., with exposure to air. These pressings were performed both by single and by double-action pressing. Several production type gang pressings were made which allowed the hot-pressing of six preforms simultaneously with exposure to the air.

III. RESULTS AND DISCUSSION

A. Wax Tumbling

Uranium powder was tumbled at approximately 45 rpm, in dry glass jars with paraffin wax shavings. The wax used was a medium U.S.P. grade, having a melting point of approximately 52°C. It was found that, although the material was at all times processed under a protective atmosphere of tank argon, it was necessary to assure the freshness of the powder at the time of processing and prevent contamination during tumbling by careful sealing of the jars. The most effective control for this was found to be ignition testing of unwaxed batch samples at various stages of processing. Spontaneous ignition of such samples when exposed to air provided a standard for the evaluation of the ignition temperature of waxed powder.

The variables investigated in this series of experiments included:

1. Powder Particle Size

- a. -20 +80 mesh
- b. -80 +200 mesh
- c. -200 + 325 mesh
- d. -12 mesh
- e. -80 +325 mesh

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2. Amount of Wax Addition

- a. 1-1/2% (by weight)
- b. 3%
- c. 5%

3. Tumbling Time

1. to 24 hours

4. Tumbling Temperature

- a. Room temperature.
- b. 45°C (below the melting point of the wax).
- c. 56°C (above the melting point of the wax).

The size of the charges was increased from 20 g to 500 g as safety considerations allowed. Table I gives a summary of the most significant data obtained. Processing and testing for ignition temperatures were carried out in accordance with procedures described above. It can be seen from Table I that increased tumbling time resulted in increased ignition temperatures for powder within a narrow particle size range. No other marked trends could be established from the available data.

Although the investigation showed that small batches of powder of a relatively narrow particle size range can be effectively wax coated by this method, the -12 mesh powder, consisting of small as well as large aggregate particles, could not be so protected consistently and effectively. The latter characteristic is possibly due to the fact that it is very difficult to coat a porous aggregate particle by a smearing action such as tumbling. The results in the case of this type of powder were generally erratic and gave no indication of culminating in a powder that was reliably safe in handling. Attempts to inhibit the pyrophoricity of -80 +325 mesh powder by tumbling 140 g batches at room temperature with 1-1/2, 3 and 5% paraffin wax also did not yield consistent results or show marked promise of success.

Several 140 gm charges of powder, tumbled with 1-1/2% wax for 1 hour, were hot-pressed, together with unwaxed standard samples, in accordance with regular uranium hot-pressing procedure. Table II summarizes the results of these pressings. The resultant densities and chemical analysis show that the waxed powder can produce a slug of acceptable density, usually slightly lower in density to slugs processed from unwaxed powder.

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TABLE I

Ignition Temperatures of Uranium Powder
Tumbled with Paraffin Wax

Mesh Fraction of Powder	Weight Of Charge gm	Paraffin Wax Weight %	Tumbling Time hrs.	Ignition Temperature °C
-20 +80	80	1-1/2	1	RT(1)
	40	1-1/2	5	RT
	80	1-1/2	16	158(2)
	80	1-1/2	24	161
-80 +200	80	1-1/2	1	RT
	80	1-1/2	5	129 and RT(3)
	80	1-1/2	16	154
	80	1-1/2	24	166
-80 +325	140	1-1/2	24	RT-122
	140	3	24	138-167
	140	5	24	RT-138
	500	3	24	RT-136
-200 +325	80	1-1/2	1	110
	80	1-1/2	5	127
	80	1-1/2	16	156
	80	1-1/2	24	157
-325	80	1-1/2	24	RT
-80	140	1-1/2	24	RT-127
-12	40	1-1/2	24	88(4)
	80	1-1/2	24	RT
	140	1-1/2	24	RT
-12	40	3	24	88-118
	80	3	24	105-125
	140	3	24	RT-128
-12	140	5	24	RT-138
-80 +325	140	1-1/2	24(5)	143(7)
	140	3	24	144
	140	-12	24	RT
-12	140	---	24 +2(6)	135(7)

(1) Room Temperature.

(2) All single numbers in column
are average of 3 samples with-
in $\pm 10^{\circ}\text{C}$.

(3) Two sets of 3 samples.

(4) Single Sample

(5) Tumbled at 45°C

(6) Tumbled at RT for 24 hrs.
+ 2 hrs. at 56°C .

(7) Average of 2 samples in
this series.

NOTE: All tumbling was performed at RT except where otherwise
indicated.

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TABLE II

Hot-Pressing of Uranium Powder, Unwaxed
and Waxed, by Tumbling

Wax - 1-1/2%
Tumbling - 1 hour
Charge - 140 gm
Standard Vacuum Hot-Pressing Procedure Used.

Mesh Fraction Powder	Density (Archimedes) g/cc	C ppm	O ₂ ppm
-20 +80 unwaxed	18.90		
-20 +80 waxed	18.80		
-80 +200 unwaxed	18.92		
-80 +200 waxed	18.89	650	260
-200 +325 unwaxed	18.90		
-200 +325 waxed	18.88		

Exploratory experiments in warm wax tumbling were carried out by tumbling 140 g -12 and -80 +325 mesh uranium powder charges with 1-1/2 and 3% wax for 24 hours: (1) at a temperature slightly below the melting point of the wax (45°C); and (2) at room temperature, followed by a 2 hour tumbling at a temperature slightly above the melting point of the wax (56°C) for the -12 mesh fraction.

The ignition temperature of the resultant waxed powder are tabulated in Table I. Although a certain inhibition of the pyrophoricity of the powder was achieved by this treatment, its possibilities for successful protection or its superiority over room temperature tumbling were not indicated.

Tumbling of the uranium powder with wax shavings as a method for successfully inhibiting the pyrophoricity of the powder were not explored further because of the development of a more successful method described later in this report.

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B. Solute Deposition Method

The second method of conditioning uranium powder so as to inhibit its pyrophoricity was essentially the coating of the powder with a paraffin wax by a solution treatment. The treatment consisted of dissolving up to 10% of paraffin wax in an inert, dried organic solvent, such as kerosene or benzene, wetting the powder by the solution under a protective atmosphere, and subsequently removing the solvent by evaporation in air or under vacuum. The wax solute remained, after the evaporation of the solvent, as a coating on the particles. In using this method, incorporating a solvent, it is imperative to assure the inertness of the solvent to the uranium powder as otherwise the operation may become very hazardous. Solvents containing halogen or polar groups should be particularly avoided. Pure, dried hydrocarbons are especially suitable for this purpose. This method of wax coating was found to be equally applicable to all mesh fractions of powder. The experiments were mostly conducted on a -12 mesh uranium powder. Benzene was found to be a satisfactory solvent, superior to kerosene due to higher vapor pressure. The dried, wax coated powder was in all cases non-pyrophoric at room temperature regardless of the wax content of the solution, which was varied from 1 to 10% by weight. A disadvantage of this process was the fact that the powder, after solvent evaporation, was consolidated as a cake. This cake, probably adequately protected in itself by the wax, after having been broken up into a pourable powder, resulted in uranium-wax aggregates containing surface lesions which transposed the bare powder surface. These bare surfaces slowly oxidized between 100 and 160°C not correlatable to other process variables causing contamination and erratic ignition temperatures. This method of wax coating a pyrophoric powder could possibly lead to satisfactory results if the wet solution slurry were dried in a rotating drum drier. This method of inhibiting uranium pyrophoricity was likewise abandoned because of the successful development of a slurry technique described below.

C. Slurry Method

1. Characteristics of the Slurry

The slurry method of inhibiting the pyrophoricity of uranium powder consisted essentially of wetting the dry powder with molten paraffin wax or a paraffin oil under a protective atmosphere. The powder remained wetted during subsequent processing until the evaporation of the paraffin during hot-pressing. All tests subsequently described were carried out with a -12 mesh uranium powder.

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The slurry was, in all cases but isolated hot-pressing experiments, processed to the finished slug while exposed to air.

Two types of paraffin were used, namely, a medium U.S.P. grade of paraffin wax and a medium viscosity white U.S.P. grade paraffin oil. In the case of the wax the mixing operation was carried out with the wax in molten condition, at a temperature slightly above its melting point. Subsequent storage was as molten or in the solid state. It was found that, while fine, -325 mesh, uranium and UH_3 powder remained, to a large degree, suspended in the slurry up to several days, the -12 mesh powder resulted in a slurry of handling properties closely paralleled by wet sand. Although no contamination of the slurry was found to occur if the slurry was processed within several hours of its exposure to air, a thin, dark brown layer, believed to be oxide, did form if exposed for more than 1 hour in molten condition. Oxidation, penetrating to a considerable depth below the surface, was also found to occur in the case of solidified slurry upon its prolonged exposure to air.

The substitution of paraffin oil for paraffin wax resulted in a simplification of processing, since it was not necessary to apply heat in order to keep the slurry in molten condition, and also improved protection against contamination. It was found that no noticeable oxide layer formed on the surface of the powder layer for 4 to 6 hours of storage. Only after longer storage did a thin oxide layer become apparent. The layer did not seem to grow in depth upon longer storage.

A cursory investigation of the flow properties of the slurry showed that 5 to 10% of oil, in excess of that necessary to wet and cover a powder aggregate, was advantageous in insuring good flow characteristics. A smooth, continuous flow of the slurry from a hopper to the die cavity was accomplished by agitating the slurry thoroughly at low speed. The transport of the slurry can possibly be effected by such means or by recirculation and/or vibration. The slurry without excess oil was found to have approximately the same apparent density as the dry powder.

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2. Preform Pressing of Slugs

The molten wax and paraffin oil slurries, described above, were fabricated into cold-pressed preforms for subsequent hot-pressing. The use of preforms in hot-pressing, rather than loose powder, makes possible a considerable cost saving in pressing equipment and dies. All loading, pressing and ejecting operations were carried out without the use of a protective atmosphere. Inconel, split insert dies, 16" long, were used for cold pressing. It was found necessary to carefully oxidize the inserts and alternately spray them with a heavy layer of Aquadag and wirebrush the sprayed surfaces in order to get effective lubrication during pressing. It was possible, however, to perform up to eight pressings* without reoxidizing when the inserts were lubricated in this manner. During the pressing, performed in a single-action or double-action manner, the greater portion of the oil or wax was separated from the uranium powder by compaction. The fluid escaping between the die wall and punches was collected in a pan and could be reused. The waste oil or wax contained, in the case of the -12 mesh powder used here, only a minimal amount of fines and superfines which are reclaimable from the waste fluid. The die, in the case of the pressing of a wax slurry, was preheated to a temperature sufficient to keep the slurry in molten condition during pressing. The pressed compact was ejected after cooling of the die. The utilization of paraffin oil, instead of molten paraffin wax, made it possible to carry out the preform pressing operation at room temperature. An additional advantage of the oil over the wax was found to be the superior storability of preforms compacted from the former type of slurry. Oiled preforms, by virtue of being completely penetrated and covered with the oil, showed considerably less tendency to surface-oxidize upon storage in air than the paraffin wax type of compacts which exhibited pronounced and penetrating surface oxidation after short periods of exposure to air, e.g., a progressively penetrating ring of oxygen-and nitrogen-contaminated powder formed, from the outside surface inward, after less than one day of storage. This phenomenon was noted to a much lesser degree in the case of paraffin oil containing preforms stored in air. The internal macrostructure of paraffin oil-containing preforms stored under the oil did not show any deterioration after more than one month's storage.

* New improvements in lubricating technique have increased the number to 20 pressings.

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Ignition tests were performed on two waxed preforms by placing them, on top of a thermocouple junction, on a heated graphite plate. The wax melted and partially evaporated before igniting at about 350°C. The powder particles exposed by the burning off of the wax oxidized slowly at first and, after several minutes of burning, commenced to oxidize vigorously causing the slug to glow.

Table III and Fig. 1 summarize the results of pressing a series of paraffin oil-uranium slurry preforms. A series of duplicate pressings at several load conditions of the above series resulted in a duplicability of within $\pm 2\%$ of the average density. This deviation falls within the error of measurement in determining the density. Although 66.8 tsi pressing load resulted in the highest preform density, 53.2 tsi pressure was used as standard in subsequent evaluation pressings in order to conform to allowable die stresses. Double action pressing resulted in superior densities as compared to single-action pressing. Fig. 1 shows that the pressure range of preform pressing used in these experiments was not sufficient to result in a complete leveling off at the higher pressures.

TABLE III

Cold-Pressing of Uranium-Paraffin Oil Preforms

1.370" in diameter

Load tsi	Density, Geometric g/cc	
	Single-Action	Double-Action
19.1	---	10.8
26.9	11.5	11.8
32.7	12.2	12.6
39.5	12.7	13.2
46.4	---	13.7
53.2	13.8	14.2
66.8	---	14.7

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3. Hot-Pressing of Slugs
from Preforms

Preforms, fabricated in the manner described above, were subsequently hot-pressed in Inconel dies, in accordance with standard hot-pressing procedure, both in vacuum and in air. The hot-pressing was, in all cases, performed at 600°C with a load of 12 tsi held 15 minutes. Generally, preforms of approximately 1.38" in diameter, (the preforms expanded about 10 mils in diameter upon ejection from the preforming die) were pressed in 1.44" diameter hot-pressing dies. In the case of hot-pressing in air, the preforms, after loading into the die, were subjected to a nominal pressure of about 3 tsi and were heated to approximately 350°C while the paraffin and its decomposition products escaped as vapors. The function of the paraffin in this operation was to provide a heavy vapor which filled the die cavity to the exclusion of air, thus preventing oxidation of the powder or partially pressed compact at elevated temperatures during the hot-pressing operation. After the escape of the major portion of the paraffin, the applied pressure was increased to approximately 6 tsi which was held to 550°C at which time the full pressure of 12 tsi was applied and maintained during the 15 minute pressing period at 600°C. The pressed slug was ejected hot or upon cooling of the die. The as hot-pressed densities of several series of slugs are tabulated in Table IV. Series A and B were performed by double-action, Series C, by single-action pressing. Series C represented a preliminary attempt to utilize existing unmodified production equipment for gang pressing of paraffin oil preforms. As shown in Table IV, it was possible to hot-press the paraffin wax- or oil-containing preforms in air with the loss of only several hundredths of g/cc in density as compared to vacuum processed and fabricated loose powder. The slugs, as hot-pressed in air, were not contaminated with the exception of a slightly higher carbon content, in excess of the contamination experienced in standard, loose powder vacuum type, operational procedure. Table V shows the extent of contamination as compared to standards of vacuum hot pressing. The basically significant feature of this table is the carbon content column. Normally, Derby uranium powder slugs contain approximately 100-150 ppm of carbon. The 500 to 600 ppm carbon content for the slug analysed indicates that some carbon was left behind in the slug by the paraffin wax additive. This would in part account for the slight loss in density of the slug of Series D as compared to the slug made from pyrophoric powder processed in vacuum, namely 18.90 vs 18.93 g/cc.

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TABLE IV

As-Pressed Densities of Paraffin-Containing
Preforms, Hot-Pressed in Air

600°C, 12 tsi, 15 minutes (-12 mesh powder)
Standard Hot Pressing Procedure (Aquadag Lubricant)

Series	Type of Slurry	Density, g/cc		Grain Size microns	Remarks
		Preform	Hot-Pressed		
A	Paraffin Wax	12.5	18.92	---	A standard run in vacuum was 18.93 g/cc
B	Paraffin Oil	12.5	18.88	---	--
C	Paraffin Oil	14.1	18.85	4-17	A standard run in vacuum was 18.93 g/cc
		14.2	18.88	4-17	
		14.0	18.75	4-17	
		14.0	18.84	4-17	Gang pressing of six simultaneous pressings in production equipment.
		14.1	18.87	4-17	
		14.3	18.85	4-17	
D	Paraffin Wax	12.5	18.90	---	This specimen was hot-pressed in vacuum.

TABLE V

Chemical Analysis of a Paraffin Wax-Containing
Preform Slug Hot-Pressed in Air

Standard Hot-Pressing Procedure in Air
600°C, 12 tsi, 15 minutes
Die Lubrication - Aquadag
Density - 18.86 g/cc
Powder -12 mesh (Derby)

Location of Sample	Weight, ppm			
	Nitrogen	Oxygen	Carbon	Hydrogen
Top Center	170	270	500	10
Middle Center	160	190	600	9
Middle Edge	160	210	600	10
Standard Vacuum Hot-Pressed Slug Without Wax.	150-175	200-250	100-150	10

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IV. CONCLUSION

Uranium powder produced by the hydriding process is extremely pyrophoric, igniting spontaneously and violently at room temperature upon exposure to air. Such powder has, to date, been handled and processed to a fully dense body only under a protective atmosphere or under vacuum. This investigation explored the feasibility of coating the powder with a paraffin wax or oil in such a manner as to enable the powder to be processed safely, without significant contamination or loss of properties and without the use of a special protective atmosphere. The methods explored included:

- (1) Cold and warm tumbling powder with paraffin wax,
- (2) Wetting powder with a paraffin wax-containing solution, followed by the removal of the solvent, and
- (3) Wetting powder with molten paraffin wax or paraffin oil.

The tumbling method did show some promise for possible development into a reliably consistent and safe method of pyrophoricity inhibition. The solute deposition method, although not conclusively successful in this preliminary investigation, indicated some possibility for success.

The last method resulted in powder, which, when handled in slurry form at all stages of fabrication, resulted in a dense body comparable in density and contamination pick-up to a standard body, handled under protective atmosphere and hot-pressed in vacuum, except for a slightly higher carbon content. The process was found to be reliably safe and applicable to all powders irrespective of particle size or other processing.

V. FUTURE WORK

Further investigation is necessary in order to go to a larger scale application of the processing of non-pyrophoric uranium powder using the slurry technique. Future work is contemplated with regard to a more detailed study of slurry mixing and pouring as well as the lubrication of dies for cold preforming work. Finally, the actual hot pressing of preforms will be explored to a greater extent. This will include investigations involving: single and double action pressing, variation of lubrication techniques and the loading and pressing of cold preforms in hot dies.

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BIBLIOGRAPHY

1. Report of Metallurgical Team Visit to Harwell and other AERE Contractors, June 16 through 26, 1953, August, 1953, WASH-343.

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Fig. 1—Plot of density vs. pressure for cold pressed preforms containing paraffin oil, aquadag lubricant, 300 tsi pressure.

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