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SEP-157

THE FABRICATION OF HOLLOW CYLINDRICAL
FUEL ELEMENTS FROM URANIUM POWER

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Metallurgy & Ceramics

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Metallurgy & Ceramics

ABSTRACT

A description is given of the die designs, dry boxes, loading cans, vacuum pots and other experimental equipment used for fabricating hollow fuel slugs from uranium metal powder. The details of equipment operation are also given, including the die lubrication, die loading, pressing and inspection procedures.

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INTRODUCTION

The purpose of this report is to describe the equipment and procedures for hot-pressing hollow slugs from uranium metal powder. Particular emphasis is placed on the equipment and procedures which differ from those used in the experimental hot-pressing of solid slugs.

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PART I. - EQUIPMENT DESCRIPTION

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a. Dies

by Carl Spradlin TISOR, date 6/22/55

All dies used to date were essentially of one basic design. The dies consist of a cylindrical 16" long die body and inserts, a solid top punch with a recess or projection matching the core rod for centering, a tapered core rod, and a lower punch through which the core rod passes. Five different setups of this basic design have been used.

The first experimental unit, see Fig. 1, was used to make Hanford size hollow slugs with 1/4", 3/4", 1" and 1-1/4" cored holes. Each size core rod required a special top punch, bottom punch, and lower plate. This unit was inefficient in that it required the use of the loading dry box to charge the dies with powder. The dies for this setup were assembled by first placing the lower plate into the collar of the extension. The lower split punch was then placed on the lower plate and each half of the punch was held together by a shear pin. The die body with the inserts in position, was carefully lowered into the collar and over the split punch. The die body and collar were then secured together by a twist lock. Next, the core rod was lowered into the die cavity until it rested on the shear pin. Before this assembly was lowered into the vacuum pot thermocouples were inserted into the die body and the lower plate.

The second experimental unit, see Fig. 2, was used for larger experimental slugs. These slugs measured 2.43" O.D.x1.38" I.D.x4" long. In this setup the core rod was pushed through a Wilson seal in the bottom of the vacuum pot. This design eliminated the need for an extension, thus permitting the use of a shorter vacuum pot and a smaller furnace. A disadvantage to this design was the difficulty in obtaining uniform pressing temperatures because of the proximity of the bottom punch to the water-cooled Wilson seal. A solid

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lower punch was used in this equipment instead of the split type used in Fig. 1. The lower plate thermocouple was completely enclosed and was not disturbed when the dies were taken out of the vacuum pot. This unit required the use of the loading dry box to charge the dies with powder.

In the third experimental unit, see Fig. 3, a die loading can identical to the Hicksville design for solid slugs, was used to charge the dies, thereby eliminating the use of the loading dry box. As the top punch was raised into the die loading can to trip the loading can sleeve the guide pin moved with it. This guide pin centered the core rod while the powder emptied into the die cavity. When the top punch contacted the core rod during compression, the core rod was pushed through the restraining ring. A restraining ring was substituted for a shear pin to prevent the core rod from falling away from the top punch when the pin sheared.

The fourth experimental unit, see Fig. 4, utilized the best features of previous designs. These features include charging the dies from a die loading can, using a solid core rod in place of a hollow core rod. In this setup special connections for the core rod to both top and bottom punches eliminated the need for shear pins or restraining rings. The core rod was connected to the top punch so that as the top punch was raised to trip the loading can sleeve the core rod moved with it. The connection of the core rod to the bottom punch prevented misalignment of these two components. Although this design has only been used with 1/4" core rods it can be adapted to larger core rods.

The fifth unit was designed to be used in the Hicksville hot-pressing equipment. This setup, (Fig. 5), is similar to the fourth setup and requires only minor modifications of the Hicksville equipment.

b. Associated Equipment

A detailed description of the following equipment, with the exception of the die loading can, is given in report SEP-110.

1. Loading Dry Box (Fig. 6)

The loading dry box is used to charge the pyrophoric uranium powder into the dies in an inert atmosphere.

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2. Die Loading Can, (Figs. 3 and 7)

The three principal components of the die loading can are the funnel, the cover, and the sleeve.

The funnel has eccentric locks to secure the cover and a water cooling jacket to protect the "O" rings. To maintain a negligible powder loss, the discharge port of the funnel is fitted onto the die extension with a loose or "Class I Fit." The inside of the discharge port has an "O" ring which seals the sleeve.

The cover consists of the powder chamber, a Wilson seal for the ram and an "O" ring for sealing the sleeve.

The die loading can is charged by inverting the assembled can and inserting a special funnel into the discharge port. After the powder has been loaded the funnel is withdrawn and the sleeve is positioned on the two "O" rings sealing the powder chamber from the external atmosphere.

The die loading can is then removed from the dry box and before it is placed on the vacuum pot, the ram and top punch are assembled to it. To dump the powder, the ram is lifted sufficiently high so that the top punch, which is screwed to the ram, trips the sleeve, permitting free flow of the powder into the die cavity.

3. Vacuum Pot, (Fig. 8)

The vacuum pot used was of the externally heated type. It was fabricated from standard steel pipe and the upper end of the vacuum pot has a flange for an "O" ring seal. A vacuum outlet of 1/2" pipe was welded under the top flange. A Veeco vacuum valve was brazed to the outlet to maintain the inert atmosphere when the vacuum pot is removed from the loading dry box.

A large Kinney Type CVD 8610 vacuum pump is used to evacuate the vacuum pot.

4. Mobile Unit, (Fig. 9)

This unit transports the furnace and vacuum pot into and out of the press and contains all the instruments and controls to regulate the furnace. A portable thermocouple vacuum gage and a potentiometer completes the instrumentation required for the hot-pressing operation.

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5. Press. (Fig. 10)

A fifty ton Dake press having a double action hydraulic cylinder is used for hot-pressing. A heat exchanger has been installed on this press to enable it to maintain full capacity loads for sustained periods.

PART II. - PROCEDURE

a. Lubrication

The dies, heated on a hot plate to about 150°F, are sprayed with a ten-to-one mixture of water and colloidal graphite. A Binks spray gun, adjusted to give complete evaporation of the water in about one second, is used for spraying. All surfaces are at least triple coated and as many as ten passes are applied to the surface which would come into contact with the powder during compacting.

b. Loading

The dies are loaded in the loading dry box or with a die loading can, depending on the setup used. The powder is made from decomposed uranium hydride and milled until an apparent density of 5g/cc or better is obtained. The apparent density specification is required to insure that the powder charge can be contained in the die cavity. Most of the slugs made to date were made from powder which passed through an 80-mesh screen. Studies are under way to determine the coarsest powder that can be used for a given core rod diameter.

c. Temperature Controls and Furnace

Two Chromel-Alumel thermocouples are used to check the die temperatures. One thermocouple is located in the die body and the other in the lower plate about 1/8" from the core rod.

The heating of the dies is controlled by three furnace thermocouples connected to Alnor Temperature Controllers. The current in each element is controlled by a Variac. Special temperature runs were made to determine the heating cycle required to heat the dies to a given temperature. The optimum pressing temperature for uranium powder was found to be about 600°C. A temperature gradient of 4°C can be maintained in the uranium compact after pressure is applied.

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d. Pressing Sequence

In the case of runs employing the loading dry box the vacuum pot is evacuated to 25 microns or better before commencing the heating cycle. The absolute pressure increases to about 200 microns after about fifty minutes in the furnace, because of degassing of the powder. By the time pressing temperatures have been reached the vacuum is back to about 20 microns and increases to 30 or 40 microns during the fifteen-minute pressing.

When the die loading can is used, the vacuum pot is filled with argon during the heating cycle and is again evacuated when the die temperature reaches 550°C. Time is solved in two ways by using the die loading can: first, the loading dry box is by-passed; and secondly, the heating cycle is shortened because the argon gas is a better heat conductor than the vacuum. When the loading dry box is used the powder and dies must be heated in a vacuum because of the getter action of the uranium powder for nitrogen and oxygen contained in the argon. In using the die loading can, the dies are heated in an argon atmosphere and, prior to dumping the powder, the system is evacuated. When the pressing temperatures have been reached, the powder is dumped into a static vacuum and allowed to heat for five minutes before being pressed. This short-time heating prior to compacting minimizes contamination of the powder.

e. Compacting Pressure

It has been found that the optimum conditions for uranium powder prepared from decomposed uranium and milled to pass through an 80-mesh sieve is about 12 tsi at 600°C for fifteen minutes. In pressing hollow cylinders it has been found necessary to increase the applied pressure in proportion to the increased side wall friction area of the core rod to obtain full density. The load in tons on the ram is given by the following formula:

$$L = p_4(D_1^2 - D_2^2) \left(1 + \frac{D_2}{D_1}\right)$$

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L = load in tons on ram
p = compacting pressure in tsi
D₁ = outside diameter of hollow cylinder in inches
D₂ = inside diameter of hollow cylinder in inches

The factor $(1 + \frac{D_2}{D_1})$ in the above equation compensates for the increased side wall frictional area of the core rod.

f. Ejection of Core Rod

In the early designs, a high rate of core rod breakage was encountered as they were ejected. This breakage generally occurred at the top of the core rod while the top punch was being removed, (see Fig. 12). This difficulty was overcome to a great extent by reversing the position of the projection and matching recess in the core rod to top punch connection. In the case of those units using the dry box, a recess in the core rod in the shape of a truncated cone assured concentric alignment and permitted the top punch to be easily removed from the core rod after pressing. In ejecting this type of core rod a special hardened punch was used to push the rod out of the slug. (See Figs. 13 and 16).

In later designs the core rods were pulled rather than pushed out of the slug. This method of ejection is advantageous in that it is less hazardous and requires lower ejection loads because of the more favorable Poisson's ratio. Core rods of this type have a threaded section on the lower end which fits into a special adapter on the pressure ram. It is believed that this method of ejection may be used for various sizes of core rods, although, at present, it has only been tried on 1/4" diameter core rods. (See Figs. 14 and 17).

With either method of ejection, it is necessary that the section of core rod within the hot-pressed slug be tapered. The taper found to be satisfactory was a function of the core rod diameter. The experimentally determined results were as follows:

<u>Core Rod Dia.</u>	<u>Taper in 4"</u>
0.250	0.010"
0.750	0.015"
1.375	0.020"

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By heating the slug containing the core rod for 2-1/2 minutes in an oven at 550°C, the load for the ejection of the core rod was further reduced because the thermal coefficient of expansion of uranium is greater than of the steel core rod.

g. Inspection of Slugs

All slugs were inspected for density, grain size, surface imperfections; and, where possible, reflectoscope data was obtained.

In computing the geometric density, the average diameter of the tapered core rod was used. This approximation of the core hole, which was actually a conical frustrum, was within 0.0005 g/cc of the true geometric density for the 1/4" diameter core hole. An Archimedes' density measurement was taken on slugs having a wall thickness of 3/16" or less.

The grain size was determined by examination of an electro-polished surface under polarized light. All slugs made to date measured 4 to 12 microns, with a few larger grains up to 17 microns.

The surface of the slugs was inspected for laminations, pits, networks, or any surface defects. The macroetch used to bring out surface imperfections was as follows:

1. Degreasing in acetone or trichloroethylene.
2. Boiling fifteen minutes in 50% sulfuric acid.
3. One-and-one-half minutes in 50% nitric acid at room temperature.

The ultrasonic testing was performed with a Sperry Reflectoscope on the ends of slugs with 3/4", or smaller, core holes. No data could be obtained from the sides of the hollow slugs because of the excessive number of reflections.

The descriptions given in this report are standard practices which have been developed at this laboratory in making hollow slugs. Deviations from these techniques are continuously being made to improve the quality of the finished slugs and to simplify the equipment and procedures used.

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PART III. - RESULTS

In pressing hollow slugs conforming to the Hanford size in outside dimensions, it was found necessary to add a factor directly proportional to the increased side wall frictional area of the core rod to obtain full density. This factor was successfully applied to slugs with different size core holes, namely, 1/4" to 1-1/4" in diameter. The compacting pressure, as a result of the frictional area factor, increased from 12 tsi to 14 tsi for the 1/4" core rod, 18.5 tsi for the 3/4" core rod, 21.4 tsi for the 1" core rod and 22.9 tsi for the 1-1/4" core rod.

The density gradient in full density slugs was virtually non-existent and variations could be detected only in the fourth significant place. It is interesting to note that in only one case the density at the bottom of the slug was found to be greater than at the top. This was the case for a slug with a 1-1/4" core hole and seems to indicate that the movement of the core rod helps to consolidate the powder at the bottom of the slug.

The chemical analysis of two hollow slugs are as follows:

<u>Slug No.</u>	<u>NAA-2</u>	<u>No. 23</u>
N ₂	0.007%	0.007%
O ₂	0.024	0.029
H ₂	0.0002	0.0032
C	0.07	0.05
Fe	0.012	0.012

On the basis of experiments performed on solid slugs it can be said that the nitrogen content of a finished slug is nearly the same or less than the nitrogen content of the raw material. The oxygen content of the finished slug is a function of the oxygen present in the raw material, the slight trace of oxygen present in the dry box atmosphere and the slight trace of oxygen in the vacuum pot which is readily picked up by the hot powder. The hydrogen content is a function of the completeness of the decomposition of the hydride in the powder preparation. The maximum hydrogen content in a hot pressed slug is 15 ppm. The carbon content is not affected by the graphite die wall lubricant, as the slugs are centerless ground to the finished dimensions. Some iron is picked up when the powder is milled.

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The number of end reflections was recorded with a Sperry Reflectoscope for various slugs. The best transmission was generally received when the testing was performed at 10 megacycles. Large internal defects, voids and regions of low density can be detected with the reflectoscope.

In making pressings with larger size powder particles, such as the -12 mesh powder, some difficulties would be encountered in pressing thin walled hollow slugs. The opening in a 12 mesh sieve is 0.055" while the wall of the hollow slug with a 1-1/4" core hole ranges from 0.066" at the top to 0.059" at the bottom. As the powder is dumped into the die cavity, the tapered core rod may concentrate the finer powder particles at the bottom of the slug, resulting in a non-uniform particle size distribution. This non-uniform particle size distribution may result in a slug with density variations. Although the largest particle from a 12 mesh sieve can enter the die cavity the probability for bridging is higher.

Five slugs with 1/4" core holes were made from -12 mesh Hicksville production powder. Their physical characteristics were equal to the -80 mesh powder.

Several slugs were sectioned for internal grain size studies. In every case the internal grain size was the same as the surface grain size and measured 4 to 12 microns with a few grains up to 17 microns. Fig. 18 is a photograph of hollow slugs of various sizes and core diameters made by the described process.

PART IV. - CONCLUSIONS

It is now possible to make hollow slugs with densities of 18.90 g/cc or better, having core holes from 1/4" to 1-3/4" with no difficulty. These slugs can be produced at the Hicksville Pilot Plant with only minor changes in the existing equipment.

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Fig. 1 First Experimental Unit.

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Fig. 2 Second Experimental Unit.

Fig. 3 Third Experimental Unit.

Note:

The powder is shown inside the die loading can held in place by the sleeve.

Fig. 4 Fourth Experimental Unit.

Fig. 5 Fifth Experimental Unit.

Fig. 6 Photograph of loading dry box and auxiliary equipment.

Fig. 7 Photograph of die loading can

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Fig. 8

Vacuum pot and cover

Fig. 9

Mobile unit

Fig. 10 Photograph of equipment during hot pressing

Fig. 11 Die body, inserts and extension

Fig. 12 Core rods, top and bottom punches
(Early designs, 1-1/4" and 3/4" diameter)

Fig. 13 Core rods, top and bottom punches
(improved design 1" and 3/4" diameter)

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Fig. 14 Tensile core rod, top and bottom punch. 1/4" diameter

Fig. 15 Ejection of die sleeves from die body.

Fig. 16 Ejection of core rod employing a compressive force.

Fig. 17 Ejection of core rod employing a tensile force.

Fig. 18 Finished hollow cylinders of various sizes

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