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PRODUCTION OF HEMISPHERES OF URANIUM AND 25 BY HOT PRESSING

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ABSTRACT

An account is given of the fabrication of beta-stage 25 nesting hemispheres used in neutron multiplication experiments. The method of fabrication, hot pressing, is described in detail, and a list of the various metal losses involved in fabrication is given. The compositions of the various lots, and composition of the various hemispheres by lot numbers are given in Table II. Because of a mistake in the isotopic composition of one of the lots of 25 (BF15), the isotopic composition originally computed for one of the hemispheres was in error.

PRODUCTION OF HEMISPHERES OF URANIUM AND 25 BY HOT PRESSING

INTRODUCTION

Shortly after the first amounts of beta-stage 25 were received, it was decided to use the first two kilograms for critical-mass experiments. These experiments first involved the fabrication of a solid 1.5"-diameter sphere made in two halves, with an 8-mm-diameter hole in the center, to house a mock fission source. As more material arrived, a shell of beta-stage 25, 1.5" inside with 1/4" wall was to be made to fit around the initial solid sphere; and finally, a 2"-ID sphere with 1/4" wall was to fit around the first shell. Hence, a larger and larger solid sphere would be assembled for tests.

The percent 25 in the first, second, and third layers was close to 73%, 72%, and 70% respectively. The masses in the same order were 525 g, 749 g, and 1246 g, making a total of 2520 g. The actual compositions and weights of the individual hemispheres are given in Table III.

HOT PRESSING TECHNIQUE

The easiest method for fabrication of the spherical shapes was the hot pressing technique originally suggested by C. S. Smith, which had been used previously for fabrication of 2"-diameter beta-stage discs and various tuballoy shapes. This method has the advantage over casting that greater accuracy of shape is possible, and no metal is left in gates or risers.

Hot pressing of tuballoy and 25 is carried out in graphite dies. The slug of metal of the correct weight is placed in the die cavity, the die plunger fitted on top, and the metal is protected from oxidizing by a slow stream of argon passing into the die cavity (see Fig. 1). The dies are placed in a 20-ton hydraulic press, and

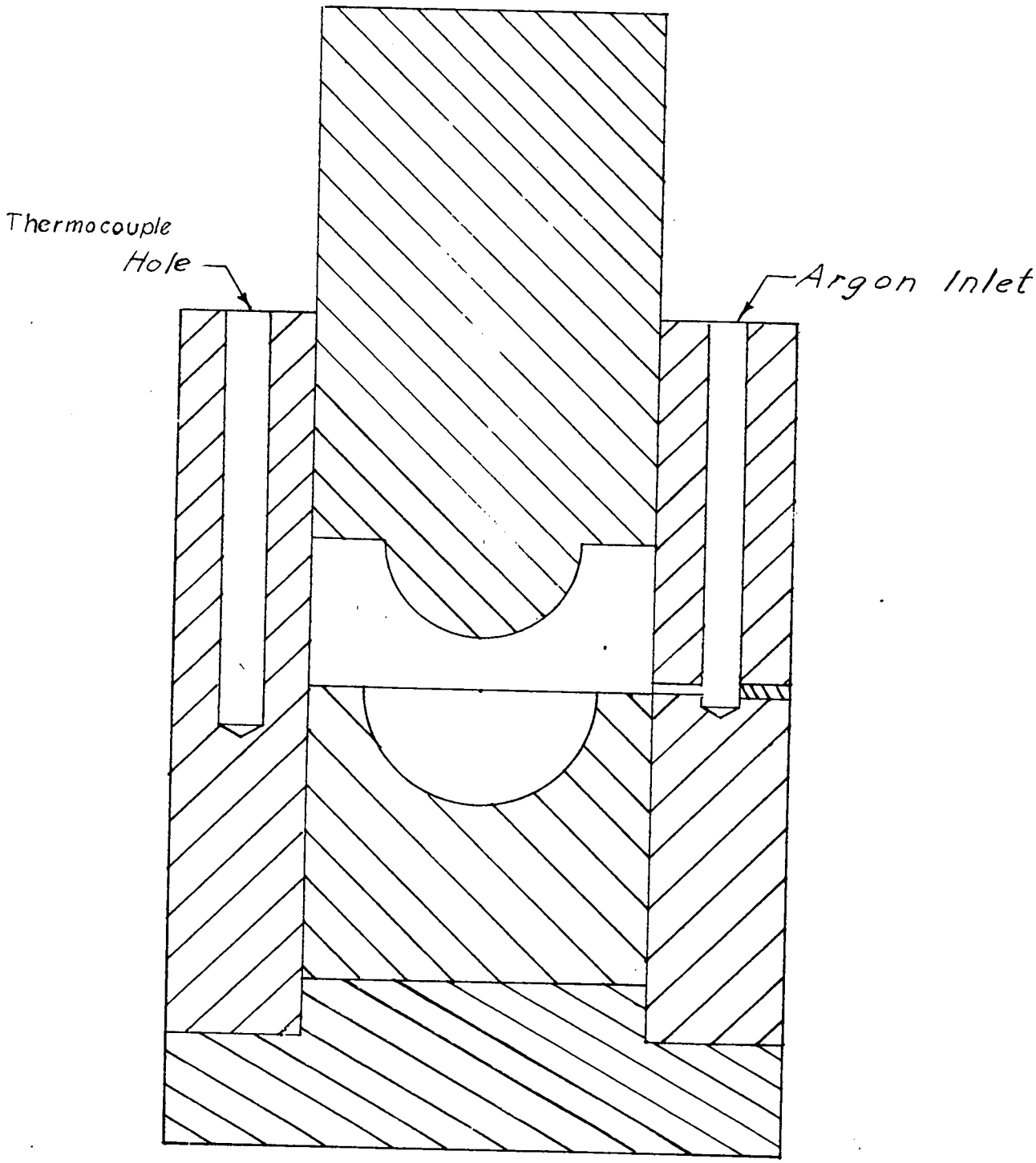

Fig I - Hot Pressing Die

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heated by a high-frequency induction coil to the hot pressing temperature, 800° to 900° C. The maximum pressure used is limited by the strength of graphite to about 5000 lbs per square inch, but fortunately the pressures required exceed this value only in the production of very thin-walled objects of considerable area where friction inhibits flow to an important extent. For example, difficulty was experienced in producing the 2"-ID hemisphere of .080" wall thickness, but a similar size sphere with 1/4" wall presented no trouble.

Some experiments have been carried out using die block inserts of materials other than graphite with somewhat encouraging results. Steatite and hot-pressed beryllium oxide appear promising.

The anticipated advantages of these materials over graphite are smoother surface and more accurate dimensions of the pressed objects; possible complete elimination of machining on the final part; in the case of some ceramic die blocks, easier fabrication than graphite, and, especially important, ability to withstand higher pressing loads.

PRESSING CONDITIONS IN PRODUCTION OF BETA-STAGE 25 HEMISPHERES

In all cases, 10 to 30 minutes are required to heat the die to the desired temperature. The outside die wall reaches a temperature of about 950°, but the inside of the die where the sample is located is only about 850° C. The pressure is applied and held for 2 to 3 minutes, and the amount of pressure varies with the shape and size of the piece as indicated below. Depending upon the size of the die, 45 minutes to an hour and a half are required to cool the die and contents down below 100° C, where the oxidation on removal into the air is negligible.

The actual pressures used for the various hemispherical shapes are given in Table I.

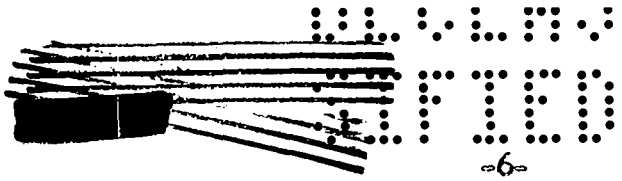


TABLE I. Pressures Used in Forming the Hemispheres

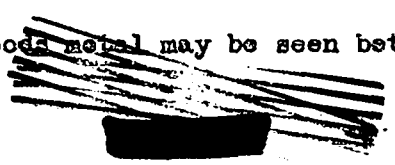
Shape	Maximum Pressure
1.5"-dia. solid hemisphere	1800 p.s.i. on projected area
1.5"-ID, 1/4"-wall hollow hemisphere	2000 p.s.i. " " "
2.0"-ID, 1/4"-wall hollow hemisphere	1600 p.s.i. " " "
2.0"-ID, .080"-wall hollow	*5100 p.s.i. " " "

*Slight cracking of die parts noted.

The shape of the metal prior to hot pressing is not very important as long as it is chunky and reasonably solid, and can fit into the die cavity. Metal which is allowed to freeze in the crucible is quite satisfactory even though such pieces invariably have shrinkage cavities or "pipe". Attempts have been made to press two or more pieces together in the graphite die with only partial success. After pressing, the metal is cleaned and a few minor machining operations are made to turn off any flash and to size the hemisphere more accurately.

In the case of the solid, central hemispheres, an 8-mm spherical hole was machined in the center for locating a source.

Fig. 2 is a photograph of the assembly as used in the final stage for multiplication experiments. Bits of Wood's metal may be seen between the layers to hold the assembly together.



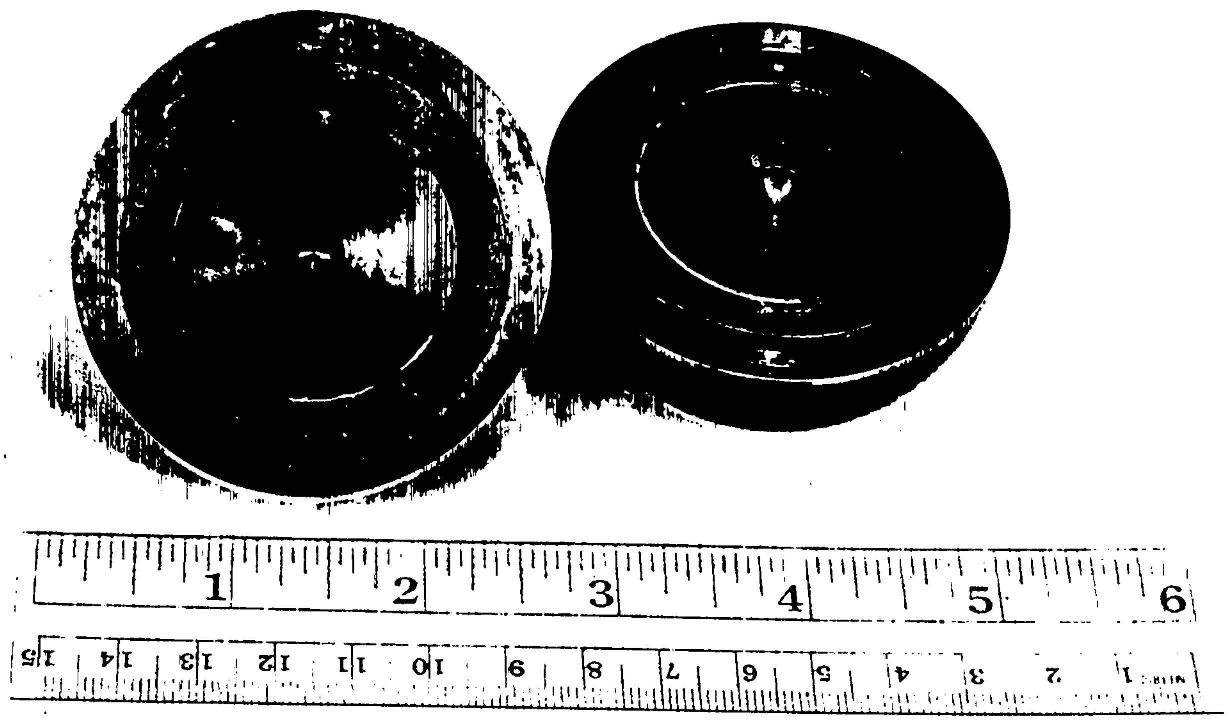


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TABLE II. Composition of the Lots, and Composition of Specimens by Lot Numbers

Lot No.	Percent 25	Hemisphere No.	Composition in Grams
BF-6	65.2	No. I	98 g BF-9 99 g BF-10 76 g BF-11
BF-7	44.4		
BF-8	70.4	No. II	100 g BF-9 91 g BF-10 82 g BF-11
BF-9	74.1		
C5-A91-112	64.0	2836	7 g BF-11 199 g BF-13 176 g BF-14
BF-10	75.5		
BF-11	69.7	2837	145 g BF-8 6 g BF-11 194 g BF-12 36 g BF-14
BF-12	73.6		
BF-13	72.3		
BF-14	72.7	2986	199 g BF-16 212 g BF-18 122 g #5071 31 g #5072 74 g #C5-A91-112
BF-15	73.8 -- 70.95**		
BF-16	73.8		
BF-17	72.5	2987	142 g BF-6 208 g BF-15 197 g BF-17 30 g #5071 60 g #5072
BF-18	73.2		
5071*	64.0		
5072*	64.0	2953	100 g #5071 76 g #C5-A91-112
		2954	66 g #5071 121 g #5072

* composite of some small early lots

** corrected number

PREPARATION OF MATERIAL BEFORE HOT PRESSING

The "biscuit" metal or reduced buttons (received from R. D. Baker) are weighed in and broken up into suitable pieces for melting. These buttons normally weigh about 250 g. The pieces are weighed and charged into an MgO or BeO crucible for melting in vacuum or argon. The weight of metal charged is essentially the weight of a hot pressing.

The metal is melted in argon rather than vacuum to minimize the amount of splattering in the furnace caused by residual calcium in the reduced biscuit metal. After attaining a temperature of about 1400° C, the high-frequency furnace is shut off and the metal allowed to solidify.

When cold, the ingot is removed from the crucible, cleaned, and inserted in the cavity of the hot-pressing die.

METAL LOSSES IN FABRICATION

The losses involved in the step of the fabrication are listed in Table III. Both the loss in grams and the loss in percent are given. Actually, these amounts of material were not lost, but were sent to recovery; but some actual loss is inevitable.

Two lots of metal sent to recovery have been assayed for a comparison between amount sent in and amount recovered. One of these was the 2"-ID, 80-mil-wall sphere, and the other the 2"-ID, 1/4"-wall sphere. The recovery was not satisfactory in the first shape, as out of 6.277 grams sent into recovery, only 4.98 grams was recovered. There was a loss of 1.297 g, or 20.6 percent. Part of the trouble here was the excessive handling during hot pressing; the piece had to be repeatedly hot pressed owing to the difficult shape (very low ratio of wall thickness to diameter).

For the heavy-wall sphere, 22.58 grams was sent in to recovery and 21.50 grams were recovered, a loss of 1.085 grams or 4.8 percent.

TABLE III. Weights and Losses in Production of Hemispheres and Shells of 25

Job	Specimen Number	Final Weight Gms	Final Isotopic Composition % 25	Losses				% Losses			
				Remelting Gms	Cleaning Gms	Hot Pressing Gms	Machining Gms	Remelting %	Cleaning %	Hot Pressing %	Machining %
1.5"-OD Solid Hemisphere	No.I	262.59	73.39	2.153	2.502	Gain 1.960	8.910	0.683	0.789	0.724	3.28
	No.II	262.41	73.51	0.583	1.792	0.000	10.870	0.106	0.654	0.000	3.93
1.5"-ID Shell 1/4" Wall	2836	374.53	72.44	1.190	3.065	0.040	3.070	0.312	0.803	0.011	0.81
	2837	374.11	72.22	3.500	2.325	Gain 0.040	2.150	0.916	0.609	Gain 0.011	0.57
2.0"-ID Shell 1/4" Wall	2986	626.72	70.11	0.692	3.625	Gain 0.125	6.695	0.109	0.569	Gain 0.020	1.052
	2987	619.16	69.0*	2.390	2.847	Gain 0.107	12.508	0.483	0.575	Gain 0.017	1.976
2.0"-ID Shell** .080" Wall	2953	181.75	64.0	0.199	2.486	0.290	1.438	0.107	1.336	0.156	0.785
	2954	178.35	64.0	Gain 0.030	4.674	0.785	3.505	Gain 0.016	2.497	0.420	1.927
Total Weights		2879.62		10.737	23.316	1.115	49.146				

* composition changed because of clerical error in composition of BF-15 at Site-X. Was originally 70.1 percent.

** not used in the assembly, but employed in a somewhat related experiment.

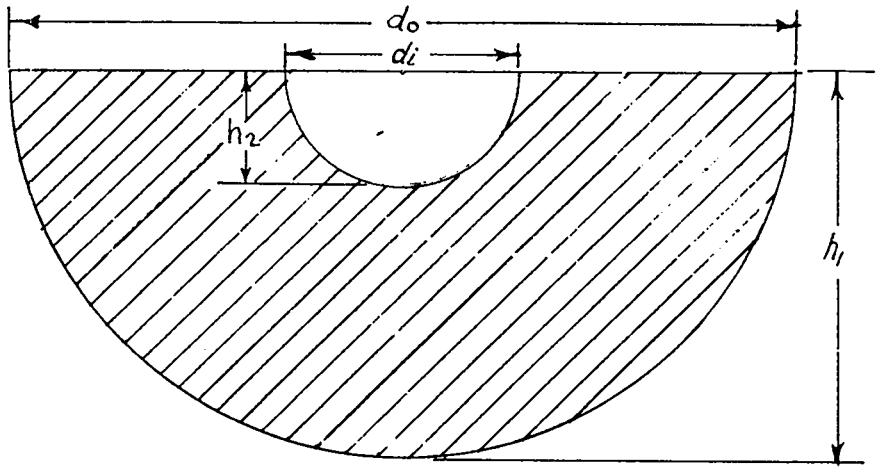
The total amount of metal handled was 2879 g, and the total "loss" in grams was 84.3 g, but this includes loss from machining of holes as well as "clean-up" machining. On this basis the loss was 3%. However, if the machining losses are discounted, and the other losses totaled, the loss was about 1%. The efficiency of the fabrication was actually somewhere between these two figures, and may be taken as approximately 2%.

Aside from machining, the largest losses arise from the cleaning operation. This in turn is necessitated by the oxidation of the metal, particularly after hot pressing.

It is believed that this source of material for recovery may be greatly lessened by the use of hard and smooth die inserts previously discussed.

In Table IV are given the final dimensions of the hemispheres; the meaning of the symbols can be seen in Fig. 3.

Drawing No. 1



Drawing No. 2

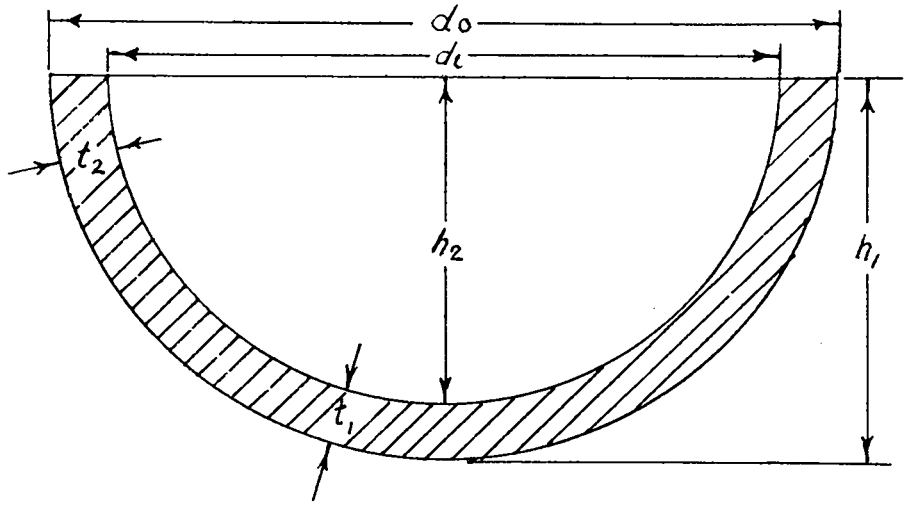


Fig. 3 - Reference Drawings for Table IV

TABLE IV. Final Dimensions (See Fig. 3 for Explanation of Symbols)

Drawing No. 1

Job	Specimen Number	d_o	d_i	h_1	h_2
1.5"-OD Solid Hemisphere	No. I	1.499" to 1.501"	0.313" to 0.314"	0.747"	0.152"
	No. II	1.494" to 1.495"	0.314" to 0.316"	0.747"	0.156"

Drawing No. II

Job	Specimen Number	d_o	d_i	h_1	h_2	t_1	t_2
1.5"-ID Shell 1/4" Wall	2836	1.999"	1.503"	1.013"	0.754"	0.254" to 0.256"	0.241" to 0.246"
	2837	1.999"	1.503"	1.008"	0.752"	0.253" to 0.256"	0.246" to 0.255"
2.0"-ID Shell 1/4" Wall	2986	2.501"	2.003"	1.273"	1.011"	0.254" to 0.258"	0.248" to 0.252"
	2987	2.499"	2.005"	1.286"	1.006"	0.251" to 0.255"	0.245" to 0.246"
2.0"-ID Shell	2953	2.180"	2.007"	1.102"	0.995"	0.095" to 0.099"	0.080" to 0.086"
	---	---	2.007"	1.106"	1.000"	0.090" to 0.092"	0.077" to 0.078"

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