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June 1945

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ABSTRACT

A description is given of a Micarta, and a silica-tube vacuum furnace together with the graphite molds used for the remelting and casting of uranium metal from powder.

1. Introduction

One of the early methods for production of uranium was the reduction of its oxide by calcium hydride as described by P. P. Alexander.⁽¹⁾ This method produced a finely divided uranium powder, which, by careful control of all raw materials, could be obtained in the requisite state of high purity. The powder was highly pyrophoric and in spite of extreme care, occasional fires occurred in the drying and subsequent handling of the material. The melting point had been variously reported as less than 1850°C (International Critical Tables) and under 1300°C and possibly less than 1200°C.⁽¹⁾

2. Earliest Experiments

Preliminary experiments on the melting of this powder were begun in March, 1942 at the request of Dr. P. P. Alexander. These experiments utilized a vacuum induction furnace which had been employed in earlier investigations of steelmaking processes.⁽²⁾ It was found that the powder could be melted readily in an alumina crucible which was placed inside a graphite crucible which served as the heating element.

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* Based on CT-539, CT-484, CT-422, CT-393, CE-345, CE-301, CE-271.

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Ingot prepared in this way and allowed to solidify in the crucible, contained about 0.5 per cent aluminum as well as considerable quantities of non-metallic matter. The metal was unmachineable and could be cut by a carborundum wheel only with difficulty. At this time beryllia crucibles were not readily available although orders were placed with several manufacturers. No melts were made in direct contact with graphite because of the reported formation of carbide under such conditions.

At this time the forms of uranium which were chosen were small blocks of four sizes: 1 inch cubes, $1 \times 1 \times \frac{1}{2}$ inch, $1 \times \frac{1}{2} \times \frac{1}{2}$ inch, and $\frac{1}{2}$ -inch cubes. It was generally anticipated that several tons of these blocks would be required. Since there was no indication that the metal was forgeable, rollable or extrudable it was decided that castings could be made of such a size that the blocks could be obtained with a minimum of cutting. For this purpose a graphite mold was constructed which contained a number of rectangular chambers formed between flat graphite plates. This mold was placed under the melting crucible in the vacuum furnace. The alumina crucible was held in a graphite crucible and both were provided with ^{bottom} holes, ~~in the bottoms~~. An alumina tube was used as a hopper-rod; a small steel rod which attached to the top of this alumina tube through a vacuum-tight silicon seal in the top of the furnace, made it possible to cast into the mold after the metal had been completely melted. The first heat of this kind was made on June 4, 1942. Six pounds of metal was cast into bars $1\frac{1}{2}$ -inch square, $\frac{1}{2}$ -inch square and $1 \times 1 \times \frac{1}{2}$ inch. The top surfaces were smooth and the edges were sharp.

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3. MICARTA TUBE VACUUM FURNACE

On the basis of the above experimental results a larger furnace of essentially the same type was constructed. Figure 1 shows a section of this furnace as it was used for several months in the production of uranium castings. The problem was greatly simplified by the discovery, which was made in another laboratory, that uranium can be melted in direct contact with graphite without significant loss as carbide. Experiments in a smaller furnace showed that the solubility of graphite in the metal at casting temperature was of the order of 0.1 per cent carbon. The use of oxide crucibles was gratefully abandoned and graphite crucibles were used as shown in Figure 1.

The Micarta tube proved to be a satisfactory vacuum chamber for the kind of vacuum obtainable with a mechanical pump. Pressures during the melting and casting operation were in the range 0.1 to 0.5 mm^{Hg}. Occasional variations up to 1 mm produced no visible effect on the quality of metal or the percentage yield. The operation of this furnace was similar to that of the silica tube furnace, a description of which follows.

4. Silica Tube Vacuum Furnace

The first of the silica tube furnaces which were built in August 1942, used a 10-inch (inside diameter) Vitreosil tube and a 6-inch (outside diameter) graphite crucible. This furnace operated quite successfully and served as the basis for the 12-inch furnace, a section of which is shown in Figure 2. Using an 8-inch graphite crucible it was possible to melt 95 pounds of metal powder or a somewhat larger quantity of lump material. By mid-November two 10-inch and three 12-inch furnaces were in operation and the production capacity of the laboratory was approxi-

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Figure 1. The Micarta vacuum furnace.

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Figure 2. The silica tube vacuum furnace.

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mately 600 pounds of castings per day. This was more than sufficient to handle all of the powder available.

5. Molds

Graphite molds machined from electrode graphite were used from the start and proved so satisfactory that no other mold materials were investigated. The shift from small cubes to cylinders weighing 6 or 7 pounds each ("eggs") greatly simplified the casting problem. These were cast in groups of three or four ingots, six to ten inches in length. A horizontal section of the mold is shown in Figure 3. It consisted of 4 bars, three inches square by ten inches long each having a longitudinal hole 2.29 inches in diameter (for a 2.25 inch casting). The inner corner of each bar was beveled in such a manner that when the bars were assembled a one-inch-square hole remained in the center. This served as the main sprue through which the metal was poured. A one-half-inch thick sprue plate was attached to the bottom of the assembly which contained the three sprues (B in Figure 3). A top sprue, T, was cut in the under side of the cover plate so that any metal in excess of 3 cylinders overflowed into the fourth. The mold was fitted with top and bottom plates of graphite and the whole assembly held together by steel bolts and straps.

This mold performed very satisfactorily when the casting temperature was 1400°C. It could be dissassembled after use and was generally good for 10 to 15 heats, except the sprue plate which was destroyed in removing the billets.

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Figure 3. Horizontal section of graphite mold.

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The shift from small cylinders to billets for extrusion or rolling again simplified the casting problem. Simple graphite molds were used for these purposes. It was noted that the pipe could be practically eliminated from the billets by placing the mold on a cold base and putting the crucible very close to or directly upon the mold. This provided a steep temperature gradient within the mold so that freezing was much slower in the top than in the bottom, resulting in an ingot of maximum soundness.

6. Operation

The powder to be charged was kept at "dry-ice" temperatures to prevent spontaneous ignition. It was charged, cold, by pouring through a funnel into the crucible inside the furnace. The cover was immediately placed on and the furnace evacuated. Power was started at 5 kw when the pressure had fallen to about 0.2 mm. Input was gradually increased, care being maintained so that the pressure did not exceed about 0.5 to 0.8 mm, the maximum power required was 30 kw. When this had been held for about 15 minutes and the temperature had reached 1400°C the metal was poured by lifting the stopper rod. The entire time of the heat averaged 70 minutes.

The crucible was reconditioned for the next melt by burning out residual uranium with a gas burner.

The principal hazards of operation were dust from the powder, fumes from cutting the sprues by an oxy-acetylene torch and smoke and dust from burning out the crucibles. This last operation was

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carried out under a special hood. The room was ventilated by a large blower and all personnel wore masks during the dusty or smoky operations mentioned.

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- 11