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UNITED STATES ATOMIC ENERGY COMMISSION

METAL HYDRIDES FINAL REPORT

By
P. P. Alexander

CLASSIFICATION CANCELLED

DATE FEB 12 1957 *md*
For The Atomic Energy Commission

H. F. Canale
Chief, Declassification Branch

July 26, 1943

Metal Hydrides, Inc.
Beverly, Massachusetts

Technical Information Extension, Oak Ridge, Tennessee

AEC RESEARCH AND DEVELOPMENT REPORT

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I. Original Work

Metal Hydrides Incorporated was the pioneer in the work of production of uranium on a commercial scale. As early as 1933, the pure metal and its alloys were produced for use in various U.S. Government and scientific laboratories.

Uranium metal was produced by the reduction of black oxide with calcium hydride. Various sources of black oxide were investigated, including oxide from the Belgian Congo and domestic oxide produced from Colorado ore purchased from the Union Carbide & Carbon Research Laboratories Inc.

The pure metal was at first produced in the form of powder which was fused into small ingots in laboratory vacuum furnaces. This was made possible by the discovery of the true melting point of pure uranium which is much lower than was reported in scientific literature, the powder fusing at a temperature as low as 1100°C, and even somewhat lower, rather than at 1700-1800°C. *

The production of commercially pure metal began on a larger scale when Columbia University started research work in the use of this metal, and placed their first order for sintered uranium in the form of discs. These were produced without difficulty and supplied to the University for Dr. Fermi's and Dr. Szilard's work. Later the conditions of the experiments were changed and larger amounts were necessary for that preliminary work. Columbia University therefore placed orders for uranium metal in the form of powder which was used to fill hollow discs and spheres. At that time it was not known what impurities in the metal will be especially detrimental and the metal was ordered without any specifications of analysis, except that the amount of occluded hydrogen should be as low as possible. This condition was easily met by degassing the powdered metal at high temperature in vacuum.

With the organization of Metal Hydrides Incorporated and the removal

* Metals & Alloys, October 1938

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of the laboratory from the Thomson Research Laboratory of the General Electric Company in Lynn, where Dr. Alexander was carrying on this work in laboratory space rented from the General Electric Company, to the Beverly factory, the work for Columbia University was started on a larger scale. The manufacture of larger quantities of this material involved problems of handling, packing, and shipping. It became evident that uranium metal in the form of fine powder is extremely dangerous material to handle. Unless it is thoroughly dried it reacts with residual moisture producing overheating of the powdered mass which occasionally results in spontaneous combustion. This difficulty was overcome by more thorough drying and storing the powdered metal, especially in large quantities in refrigerated containers.

The use of powdered metal in experimental work, however, was only temporary, and the scientists doing this work at Columbia University came to the conclusion that fused metal which would give much higher density than powdered metal even tightly packed, would be more desirable. The experiments therefore were renewed on the production of fused metal.

In the meantime it became evident that still larger quantities of this metal would be needed, and a contract was made with the Bureau of Standards to produce 7,000 pounds of commercially pure metal in powder form. The price at which the metal in small quantities was supplied was reduced from the prevailing market price of \$50.00 per pound to \$20.00 per pound when it was supplied to Columbia University, and then, in view of the great urgency for this material and the limited appropriation available for this initial work, it was agreed that the price for the first seven thousand pounds would be reduced to \$5.70 per pound which of course meant that Metal Hydrides would produce this material at a loss. Yet the Directors of the Company knowing the importance of this work gladly agreed to accept this order at such a nominal price, hoping that this contribution would allow the scientists to achieve results which might be of importance to national defense.

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It should be stated that at that stage of the work Metal Hydrides Incorporated received great help and cooperation from Columbia University and the Bureau of Standards without which this work could hardly have been undertaken.

II. Production of Metal from Canadian Black Oxide

The work was started on the production of this initial order and some 500 pounds of metal was produced by using the Canadian black oxide purchased by the Bureau of Standards from the Eldorado Gold Mines Limited of Canada.

In the meantime the scientists found that in addition to hydrogen, another impurity, boron, was extremely detrimental and that its presence in the metal would prevent them from obtaining the expected results. It was further found that boron was present in minute quantities in Canadian black oxide, but was absent in the commercial grades of calcium metal produced in France which were used on previous work by Metal Hydrides Incorporated. However, with the stoppage of imports of French calcium, the domestic calcium produced by the Union Carbide & Carbon Corporation had to be used, which was found to contain large amounts of boron. It was decided that before going any further with the production of metal, it would be necessary first, to make arrangements for the purification of the Canadian black oxide by leaching it in acid and obtaining by some means calcium free from boron.

Through arrangements with the Eldorado Gold Mines Ltd. and the Bureau of Standards, Mr. Pochon, the chief chemist of the Eldorado Gold Mines Ltd. developed a grade of black oxide which was much lower in boron.

At the same time Dr. Rodden of the Bureau of Standards found that by distilling commercially impure calcium in vacuum it was possible to eliminate practically all the boron and so obtain material which would be entirely satisfactory for use as a reducing reagent in the production of pure uranium. With the help of the Bureau of Standards the distillation plant was set up at the plant of Metal Hydrides Incorporated, for the production of distilled

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calcium metal on a commercial scale.

Metal Hydrides Incorporated found that distilled calcium could be converted into calcium hydride in the same operation. This eliminated additional expense and considerable danger in handling freshly distilled calcium. The distillation plant started production of distilled calcium hydride at first on a reduced scale which was gradually increased to several hundred pounds per day. The first small lot of purified black oxide from Canada was reduced with pure distilled calcium hydride and the produced metal was sent to the Bureau of Standards for analysis. It was reported by Dr. Rodden that the boron content was less than one half part per million and the rare earths were entirely absent, which indicated that by these means it would be possible to produce material of satisfactory purity.

III. Beginning of the Work for the O.S.R.D.

In the meantime the scientists found that still larger quantities of material would be needed for their experiments. A contract was negotiated, therefore, with the Office of Scientific Research and Development. It was insisted that since this contract was of an experimental nature, it should, therefore, be of a non-profit type. However, the Office of Scientific Research and Development agreed that in view of the unusual nature of this material no analysis would be specified and that all actual costs for the production of this metal would be taken care of, and that the contractor, although he would not make any profit, would be guaranteed "to come out whole regardless of any contingencies which might be encountered by him in fulfilling this contract." A sum of \$400,000 was specified in the contract for the rearrangement of the factory facilities, the purchase and installation of machinery, as well as for the production of metal in whatever quantities could be obtained.

The design of the machinery as well as the factory facilities were left entirely to the contractor. At that time no one could give a definite recom-

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mentation as to what type of machinery would be needed for the production of this metal on a larger scale. The installation of the machinery under present conditions took much longer than under normal conditions due to the extremely slow deliveries of various pieces of apparatus by the subcontractors.

The first contract with the Office of Scientific Research and Development expiring on June 30, 1942, was renewed to November 1, 1942, and a clause was inserted stating that besides bare costs, the contractor would receive a fixed fee representing 7% of the estimated cost. The estimated cost as calculated by the representatives of the Office of Scientific Research and Development was set at \$15.00 per pound. The analysis was still not specified in the contract, yet certain specifications for the purity of the metal were in the process of being drawn up and soon after the contract was put into effect a list of various elements and the limits which should not be exceeded became available.

IV. Casting and Sintering of Metal

The original work of Metal Hydrides Incorporated demonstrated that uranium powder could be melted very successfully in vacuum at the comparatively low temperatures of 1100-1200°C. The melting was carried out in small laboratory furnaces and necessarily was limited to the production of only very small ingots. However, an investigation was started to find a more rapid method of melting, and the powdered material was sent to several laboratories equipped with high frequency vacuum furnaces.

The first results at the Bureau of Standards and in the General Electric Research Laboratory in Schenectady were negative, and opinions on the practicability of using the high frequency method were divided. Metal Hydrides Incorporated wanted to continue the investigation and find some laboratory where the melting of uranium powder in a high frequency furnace could be tried out again; whereas other interested parties held the opinion that the fusion should be made in the resistance heater type furnaces.

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In the meantime samples of powdered uranium metal were submitted to the Massachusetts Institute of Technology and arrangements were made at the Department of Metallurgy for Dr. Chipman to attempt to fuse this metal in a high frequency vacuum furnace. In spite of several failures in the past in other laboratories to achieve such results, Dr. Chipman succeeded in fusing the very first sample of metal into a well-formed disc of 1" in diameter by using a special technic, namely by placing the powdered metal in a graphite crucible. The previous failures were due to the fact that attempts were made to fuse powdered metal in crucibles made of the usual refractories. In this case the heat was generated only in isolated particles of the metal. It took too long to bring the powdered mass to a temperature sufficiently high for fusion, and in spite of vacuum the mass was partly oxidized by remaining traces of air before the desired temperature was reached.

Professor Chipman's technic was to place the powder in the graphite crucible which being a conductor was heated first. The powdered metal was heated both by the generation of heat inside of the isolated particles as well as by radiation from the incandescent graphite crucible. The fusion was effected in a very short time. Furthermore, the incandescent graphite crucible which was also provided with a graphite cover, was acting as a very efficient deoxidizer. Oxygen leaking through the furnace was immediately combining with incandescent graphite with the result that all the oxygen, before it could reach the uranium particles, was combined with carbon forming carbon monoxide.

Professor Chipman at that time had at his disposal for these experiments only a small high frequency vacuum furnace giving ingots of a few hundred grams each. Arrangements were made immediately between Metal Hydrides Incorporated and the Massachusetts Institute of Technology, and Professor Chipman proceeded to adopt his larger high frequency furnace for the above work. Metal Hydrides Incorporated furnished a more efficient and larger

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capacity vacuum pump and made a subcontract with the Massachusetts Institute of Technology providing the necessary funds to proceed with this work without delay.

The necessary preliminary experiments required a certain time to determine conditions under which larger ingots, free from blowholes and pipes could be produced. Professor Chipman therefore was confronted with the problem not only of fusing the powder but also of casting the metal in vacuum into large ingots.

In the meantime metal for the initial experiments in Chicago was needed so urgently that it was decided temporarily to use another expedient of consolidating powdered metal into small solid billets. The sintering of the powder which was successfully carried on by Metal Hydrides Incorporated in the production of the first solid discs for Columbia University was resumed. Arrangements were made with the Massachusetts Institute of Technology to get a larger hydraulic press for pressing the powdered metal at 70 tons per square inch pressure into 1" cubes. Facilities were provided by the Massachusetts Institute of Technology and the work was started under the direction of Dr. Wulff.

The pressed cubes were transported back to Metal Hydrides Incorporated in Beverly where they were sintered into solid blocks. The density specified by the Chicago group was not less than 15, and the first series of pressed cubes after sintering were of even higher density, in some cases reaching 17. These cubes were well-formed and after sintering were resisting successfully any further oxidation. A group of scientists were sent from Chicago and the work of pressing at M. I. T. was organized on a very much larger scale. Facilities were provided to press up to several hundred pounds of cubes per day which on the following day were transported back to Beverly and sintered into solid blocks.

At that time, however, Metal Hydrides Incorporated was requested to

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porous so that during the sintering the material was much more readily oxidized by the traces of air present in the vacuum furnace, giving material containing quite a large amount of oxide and nitride. Furthermore, on account of the presence of microscopic voids and channels through the sintered compacts it was subject to further oxidation when left standing in air, with the result that some of the compacts under especially unfavorable conditions such as high humidity and high temperature of the air were gradually oxidized to such a degree that the generated heat was sufficient to ignite the compact. Once the compact was ignited, on account of its spongy structure it was burning until it was transformed entirely into black oxide. Furthermore, the heat conductivity of the sintered material produced from the brown dioxide was comparatively low and not suitable for use in further experiments. The pressing of the compacts therefore was discontinued as soon as Professor Chipman succeeded in bringing his method of fusion and pouring and casting in vacuum to the desired degree of perfection.

The total amount of pressed and sintered cubes produced during this transition period was 3,868 pounds.

V. Reduction of Brown Dioxide

The reduction to metal of the brown dioxide by the calcium hydride process, however, has certain advantages. It contains less oxygen and therefore involves a somewhat smaller amount of calcium hydride. The difference however, is not very great and does not affect materially the cost of reduction. The important advantage, however, is the fact that the reaction was proceeding much more quietly without a sudden evolution of heat as in the case of the black oxide.

The disadvantage of using the brown dioxide is that the resulting powder is not uniform. It consists of a mixture of minute particles of less than one micron in diameter, a series of solid particles of larger dimensions and very large particles in the form of hollow spheres.

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The leaching of such material was a complex problem since the conditions had to be regulated to leach quickly and without excessive oxidation the particles of micron size which required only a short time. The larger particles, surrounded by larger amounts of calcium oxide, required a longer leaching time. Finally the hollow spheres could not be leached effectively at all in a short time with dilute acids since the calcium oxide held inside of the hollow sphere was more or less protected from the action of the acid and was not removed from it but merely transformed into calcium hydroxide which completely filled the hollow spheres.

These large particles containing entrapped calcium hydroxide could not be dried thoroughly and retained the moisture for several days. During this period the entrapped calcium hydroxide was reacting with the metal with the generation of heat. This would invariably produce overheating of the powder and the ignition of same.

The leaching operation was regulated to be suitable for the bulk of the material, that is, the medium size solid particles. The very fine particles of course were partly oxidized since the time was too long, and they had to be separated by decantation, since the partly oxidized particles are extremely pyrophoric and on several occasions ignited the whole charge. The very large particles consisting of either hollow spheres or filigree-like structures had to be separated by screening the wet powder. The percentage of this material was on an average about 4% of the total weight of the metal. It could not be melted and was too dangerous to be left standing in air and therefore had to be burned to black oxide and sent for retreatment. The necessity of the separation of the very fine particles as well as very large ones was reducing considerably the efficiency of the process. Yet it had to be done to eliminate the danger of fires which at first were quite frequent. On several occasions the produced powder, if not treated as above, spontaneously caught fire even when still wet and almost immediately after discharge

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from the leachers, during the drying operation, or at times when apparently perfectly dry before it could be melted into solid ingots. Several serious fires occurred in which our men were injured, and several hundred pounds of material burned. These accidents were reduced to an absolute minimum by adopting the above technic of separation of very fine and very large particles from the bulk of the metal.

However this separation entailed a loss of about 20% of metal. Most of the 20% loss was recovered as oxide and sent to the Mallinckrodt factory for reprocessing. The exact amount of loss could not be estimated since the fine material, decanted and collected in the catch basin in the yard, periodically was spontaneously igniting. When this occurred a column of flame about 6 feet in diameter was shooting up to a height of 20-25 feet. An appreciable percentage of burning particles of fine metal was therefore projected up into the air and scattered. During the ignition of the well, the heat was extremely intense and was almost unbearable 30-50 feet away from the flame. It is estimated that the temperature of the burning uranium powder is about 200-300°C, higher than that of a burning thermite incendiary bomb.

In conclusion it may be stated that the purified brown dioxide from a certain point of view was more suitable for reduction by calcium hydride than the black oxide, yet its use involved far greater danger in handling and due to the necessity of discarding very fine metal and coarse particles, was reducing the efficiency very considerably.

VI. Distillation of Calcium

Calcium hydride produced from distilled calcium was used exclusively for the reduction of the brown dioxide since the crude calcium or recast calcium still contained boron in amounts far above acceptable limits. The distillation of calcium and the production of pure hydride was carried out as follows.

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The purity of the calcium hydride produced by this method was very high. The percentage of boron in most of the runs was less than half of one part per million.

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VII. Reduction of Trialloy

The pure distilled calcium hydride was removed from the upper part of the iron cylinders by means of a cold chisel, an operation which did not involve any difficulty since calcium hydride is a brittle chalk-like material. Then the obtained pure calcium hydride was crushed in a jaw-crusher to a mixture consisting of fine powder and particles of about $\frac{1}{4} \times \frac{1}{4} \times \frac{1}{2}$ ". During the first months of operation and before the ordered Stimson intensive mixer was installed, both the crushing and mixing were done with the apparatus previously used by Metal Hydrides Incorporated for other work, and not entirely suitable for the above purpose. However, the results were sufficiently satisfactory. The mixture of brown dioxide and calcium hydride was placed in an iron cylinder which was placed in a nichrome retort of identical design as those used for distillation. (See Plate II)

During the first month and before it became known that titanium was a detrimental impurity in the final metal, the baffle, filled with titanium oxide, was placed on top of the iron cylinder containing the mixture. Titanium dioxide was used as an efficient heat baffle as well as an absorbent of sodium.

Calcium hydride even after distillation contains a certain amount of sodium. During the heat treatment of the charge the sodium evaporates and if the temperature is still low does not react with uranium dioxide. It diffuses through the charge out of the iron cylinder and has a tendency to condense in the cooler parts of the nichrome retorts, clogging up the pipes and even completely sealing the safety valves, which renders them ineffective and the operation dangerous. Furthermore, if a considerable amount of sodium should distill out from the charge and condense in the upper part of the retort, on opening, the distilled sodium would invariably catch fire. On some occasions such a fire in the retort could extend to the charge of uranium with the resulting complete loss of valuable material. However, the presence of titanium oxide

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spread in the shallow container in the upper part of the retort was absorbing most of the distilled sodium. The sodium vapor had a tendency to diffuse toward the titanium oxide and combine with it, producing titanium sub-oxide and sodium dioxide.

This was an effective method of absorption of sodium vapor and a patent application to cover this feature was filed. However, later it became known that titanium was an undesirable impurity, and the use of this metal or its compounds had to be eliminated entirely from the process. Therefore, the use of titanium oxide as an absorber was discontinued. Fortunately, the percentage of sodium in the produced calcium hydride was very much less during the last months of operation, which eliminated the necessity of the use of titanium oxide absorbers.

VIII. Pyrophoric Properties of the Powdered Metal

Uranium in the form of very fine powder is subject to oxidation by air or moisture at a comparatively low temperature. For instance, it slowly reacts with water at temperatures above 20°C. If a large amount of powder is being acted upon, the heat of reaction is sufficient to raise the temperature gradually until it ignites even under water. When this happens, the water in immediate contact with the powdered metal decomposes into oxygen and hydrogen with a resulting explosion. On several occasions when it was attempted to store uranium powdered metal under water, after a few hours the contents of the vessel usually exploded. Therefore, uranium in the form of fine powder must be kept always in a perfectly dry atmosphere of such neutral gas as helium or argon.

The dry powder also reacts with air at a comparatively low temperature and if large masses of powdered metal are exposed to air they gradually oxidize at least on the surface to black oxide with the consequent generation of heat which under favorable conditions leads to the ignition of the powder. Furthermore, it was found that various samples of powdered uranium react dif-

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thoroughly degassed. The charge after being removed from the furnace was in the form of a sintered clinker and had to be removed from the iron cylinder by breaking it with the air chisel. Then this material was passed through the jaw crusher and crushed to a rough powder consisting of fine material and pieces up to $\frac{1}{2}$ " in diameter. Before leaching this material an analysis was made on this product. If it was reported that boron was within the acceptable limits, which usually was in the order of $\frac{1}{2}$ part per million, the charge, now consisting of fine particles of trialloy metal embedded in pure calcium oxide, was placed in the hopper and gradually shaken into the leaching cylinder containing a very dilute solution of acetic acid. (See Plate III)

All previous work by Metal Hydrides Incorporated was done by using a glass-lined leaching tank which was a very convenient apparatus for this operation. However, when work on a larger scale was undertaken on the contract for the Office of Scientific Research and Development, a recommendation was made not to use glass-lined leaching tanks, since it was feared that the material might be contaminated with the boron usually present in the glass linings of the tanks. At a later date it was proven by careful analysis that no such contamination does occur.

The rubber-lined tanks might have some advantage, but in the case of uranium have proved to be distinctly undesirable from the point of view of safety. During the leaching of the material a considerable amount of hydrogen was evolving from the charge, which was forming a layer of bubbles on the surface of the leaching solution. On some occasions these hydrogen bubbles were igniting. This could lead to serious fires and even explosions. On one occasion a fire in the leacher spread from the hydrogen bubbles to the uranium powder present on the surface of the leaching drum above the water and ignited the rubber linings of the tank. Fortunately the men in charge of the leaching operation took prompt measures to extinguish that fire before it became serious. If it were allowed to spread to the main charge of

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some 300 lbs. of uranium powder which was below the surface of the water, it would ignite that charge with the resulting reaction with water and explosion of the tank.

The leaching of uranium powder in quantities of several hundred pounds is a distinctly dangerous operation and can be undertaken only by a well-trained leaching crew thoroughly familiar with all the dangers of such an operation as well as with the necessary fire-fighting measures which must be put into effect without a single second's hesitation.

The fires in the leachers lately have not been very frequent and in every case have been instantly extinguished by using fine sprays of cold water. The safety of the leaching operation depends on a well-trained crew who know their job and do not hesitate to act.

The concentration of acetic acid in the leaching operation never exceeded some 2% by weight. A greater acid concentration is likely to oxidize the material. The temperature of the water is also an important point and has to be kept at the lowest possible limit and should never be allowed to rise above 35°C. Above that temperature the uranium powder begins to react with water with a considerable evolution of heat and the production of hydrogen. This reaction may accelerate automatically until the whole charge of some 200-300 pounds of uranium will react with water with a resulting explosion. A constant vigilance of the temperatures in the leaching tanks is a primary consideration for the safety of the operation.

As has been stated, the very large particles as well as extremely fine particles give pyrophoric material and therefore have to be separated from the main charge. The separation of the fine particles was effected by decantation. The agitation in the leaching tank was stopped and the main charge of the powder was left to settle. Very fine particles, however, still remain in suspension in the water and were decanted from the leaching drums through the special drain outside of the factory, and left to settle in a special settling

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well. These particles, when accumulated in appreciable amount in the well, usually ignited spontaneously, once every few days. However, since such fires were occurring outside of the factory they were not presenting any danger.

Usually a column of flame six feet in diameter (the diameter of the settling well) and 25-30 feet in height, was rising and burning for a period of thirty seconds, after which the flames were dying down. The flames consisted of burning hydrogen produced by the decomposition of water by burning uranium.

The remaining oxide in the well was scraped periodically and shipped to the Mallinckrodt factory for reprocessing. The larger particles were separated from the main charge by passing the whole charge when wet through a screen. These larger particles could not be melted or stored without danger of spontaneous combustion. They were burned periodically outside of the buildings of the factory and the oxide returned to the Mallinckrodt factory for reprocessing.

X. Drying

The wet uranium powder, separated from the very large and very fine particles, after thorough washing on the screen with filtered cold water, was removed to a drying room where after spreading in a very thin layer on metallic trays it was placed in the drying ovens provided with a continuous circulation of dry nitrogen. The dry nitrogen was passing over the surface of the uranium powder, then through moisture-absorbing drums filled with silica where the moisture was removed and back again into the drying oven as shown on Plate IV. The drying operation required twelve hours on an average and was conducted at nighttime. Every charge, therefore, was dried by the following morning and was ready to be sent to the foundry for melting. Plate V.

The storage of powdered uranium was found possible at low temperature.

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Therefore, cans filled with powdered metal were stored in special refrigerators and cooled to a temperature of -15°C until such time as they were removed to the furnace room for remelting.

XI. Melting

The uranium powder, as stated before, had been melted in vacuum by Metal Hydrides Incorporated when producing this metal for various laboratories since 1936. However, the melting and casting of this material in large quantities requires an entirely different technic. The problem was solved by Professor John Chipman, who devised a method of fusing uranium powder in high frequency furnaces in graphite crucibles. After the development of this method of fusion and casting at the Massachusetts Institute of Technology a special foundry was equipped at Metal Hydrides Incorporated in Beverly, where powdered material can be melted at the rate of about one ton per day on a three shift schedule.

The method consists essentially, as shown on Plate V, of placing the powdered metal in a graphite crucible provided with an opening in the bottom. This bottom opening normally is closed by a graphite rod extending above the surface of the crucible and even protruding outside of the furnace through a specially designed vacuum tight sleeve. Below the graphite crucible there is a graphite mold to which the molten metal is admitted when the graphite rod is lifted, allowing the molten metal to run down. The heating and melting of the powdered charge usually requires about one hour. The temperature of the metal observed through a glass window in the upper part of the furnace by an optical pyrometer, is raised to 1480°C . Then the power is switched off and the molten metal is allowed to cool down to 1420°C , which is the pouring temperature of the metal.

The metal poured into the mold solidifies very quickly and can be removed from the mold while it is still quite hot. Uranium metal in solid state oxidizes only on the surface.

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The produced ingots are removed from the mold and allowed to cool in air to about room temperature after they are cleaned from the outside scale and out into the desired size. The lower protruding part of a smaller diameter is cut off and used for taking samples and tests. The chemical analysis of each ingot is ascertained before it is shipped to Chicago.

XII. Conclusions

Metal Hydrides was the pioneer in the production of this metal and supplied it to all the laboratories interested in this research before other methods for its production were developed. The conditions with regard to the analysis, physical form or quantities have been changing, as in every research project, continuously. Therefore, Metal Hydrides Incorporated went through various stages of production of this metal as requested by the interested parties.

At first the metal was produced in small sintered discs about 1" in diameter. Then it was produced in small fused ingots of 2" x $\frac{1}{4}$ " x $\frac{1}{4}$ ". Later larger amounts were supplied in powder form. Later the sintered cubes were produced and finally all the metal was cast into ingots of various sizes which varied from time to time from 1" to 6" in diameter, and height was varied from 1" to 11".

The chemical analysis was also changed in accordance with the progress of the research by the physicists experimenting with this material. At first standard black oxide shipped directly from Canada was used in experiments with the only precaution of eliminating hydrogen from the produced metal. Gradually it became known that other elements should be eliminated from the metal. The latest specifications list about fifty various elements which must be kept below specified limits. The most difficult problem was the elimination of boron which at first was as high as 5-10 parts per million. Gradually it was reduced to below one part per million and most of the metal which Metal Hydrides Incorporated is now producing has a boron content of

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only .1 to .5 of one part per million.

The price of production at first was very high due to losses through fires, accidents and other inevitable contingencies. However, gradually it was brought down to a fairly low figure.

In the attached Appendices are given the representation analysis and various other data accumulated during the production of this metal.

J. H. Alexander

President and General Manager

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The Contract

On April 20th 1942, a Contract No. OEMsr-333 was granted by the Office of Scientific Research and Development to Metal Hydrides Incorporated for the purpose of starting the production of Uranium Metal on a larger scale.

Metal Hydrides Incorporated was producing at that time uranium metal on a semi-laboratory scale. The contracting parties recognized that "Uranium has never been produced in quantity prior to the effective date hereof and that its manufacture will involve great engineering difficulties and considerable danger from uranium poisoning, fire, and explosion;" that "It is impossible for a contractor to insure himself against all contingencies because of the above mentioned factors;" and that "The Government is willing to assume the actual cost of all the work hereunder and all the risks thereof, so that the contractor will come out whole, regardless of contingencies."

A sum of four-hundred thousand dollars (\$400,000) was appropriated to cover all the expenses for purchase and installation of the necessary equipment, alterations in the plant to accommodate the said equipment, and to reduce fire and explosion hazards. This sum was also to cover the expense of operation of the plant and the production of as much uranium metal as possible.

Due to an extremely slow delivery of the ordered machinery and a number of changes in the specifications on the purity and physical state of the required metal, the starting of regular production work was delayed and it was necessary to negotiate on July 27th an extension of the time for the above contract until October 31, 1942.

After considerable delays and several mishaps and accidents, the production was finally started.

By October 31st, 7460 pounds of metal passed the acceptance tests and were shipped to Chicago.

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The total expenditures on this contract amounted to \$369,300.26, which is well within the appropriated sum.

The expenditures for various items were as follows:

Table A

Vouchered by Metal Hydrides Incorporated

Machinery & Equipment	\$106,670.71
Other Supplies & Expense	100,804.03
Direct Materials	43,229.31
Direct Wages	31,400.41
Prorated Expense	24,885.63
M. I. T.	9,850.86
Travel	992.05
Fixed Fee	<u>7,803.98</u>
	\$325,636.98

Direct Payments by O.S.R.D.

Materials (Direct)	18,993.83
Other Supplies & Expense	<u>24,669.45</u>
Total Cost of	<u><u>\$369,300.26</u></u>

All the expenditures on this contract were checked by the Government representatives. The vouchers checked and certified by Lt. Col. Vanden Bulck were forwarded to the Office of Scientific Research and Development.

Most of the expenditures have been already reimbursed to Metal Hydrides Incorporated, and the equipment and remaining supplies purchased under this contract were transferred to the U. S. Engineers.

This pioneer work made it possible for Metal Hydrides Incorporated to continue this work for another branch of the U. S. Government and to furnish over 70,000 pounds of cast metal of a purity which no one thought possible.

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RESULTS IN PERCENT

M. H. Number	918	916	957	951	954	107	762-3-5
Insol.	0.06	0.06	0.62	0.21	0.14	0.36	0.16
SiO ₂	0.005	0.006	0.006	0.006	0.006	0.09	0.06
Sulfides	0.01	0.01	0.01	0.01	0.01	0.01	0.025
CO ₃	0.04	0.03	0.05	0.04	0.05	0.08	
Fe ₂ O ₃	0.02	0.02	0.07	0.05	0.08	0.05	
TiO ₂	0.04	0.04	0.47	0.12	0.04	0.15	
Nitrogen	0.21	0.18	0.19	0.14	0.23	0.27	
Chloride	<0.003	<0.003	<0.003	0.003	<0.003	0.003	
Hydrogen	0.006	0.004	0.003	0.003	0.002		
Carbon	0.03	0.05	0.04	0.03	0.03		
R. Earth	<0.001	<0.001	<0.001	<0.001	<0.001		0.001
Boron (in p.p.m.)	<0.5	<0.5	<0.5	<0.5	<0.5	0.5	0.8

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PLATE I

CALCIUM DISTILLATION AND HYDRIDING FURNACE

- a) Calcium Carrots
- b) Distilled Calcium
- 1) Furnace, brick lined, steel shell
- 2) Retort (Special Alloy) 40" x 18 1/2" od
- 2a) Retort Cover (" ")
- 3) Inside Cylinder (steel) 33" x 16 1/2" od
- 4) Heat Baffle and Sodium Trap
- 5) Anti-Splash Baffle
- 6) Burner (tangential) oil
- 7) Thermocouple
- 8) Pyro
- 9) Safety Valve Line
- 10) Gauge
- 11) Hydrogen and Vacuum Line

METAL HYDRIDES INCORPORATED

Beverly, Massachusetts.

Drawn approximately to scale

A.C.H.Jr. July 20, 1943

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PLATE II

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REDUCTION FURNACE

- a) Charge: Tri Alloy Oxide and Calcium Hydride
- 1) Furnace: brick lined, steel shell
- 2) Retort (Special Alloy) 40" X 18 1/2" OD
- 2a) Retort Cover (" ")
- 3) Inside Cylinder (steel) 18" X 16 1/2" OD
- 4) Heat Baffle: fire brick in steel shell
- 5) Sodium Trap
- 6) Burner (tangential) oil and gas (2)
- 7) Thermocouple
- 8) Hydrogen Relief
- 11) Safety Valve Line
- 10) Pyod
- 11 Gauge
- 12) Hydrogen + Vacuum Line

METAL HYDRIDES INCORPORATED

Beverly, Massachusetts

Drawn approximately to scale

A C H Jr July 20, 1943

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PLATE III

- 1) Acetic Acid Supply
- 2) Syntron Feeder with Charge
- 3) Drum steel shell, rubber lined
- 4) Manhole
- 5) Plug for hose attachment to empty
- 6) Hydrogen bubbles rising through acid and water
- 7) Supports
- 8) Charge
- 9) Vacuum Filter
- 10) Hydrogen Exhaust
- 11) Drive, chain and reduction gear

LEACHING EQUIPMENT

METAL HYDRIDES INCORPORATED

Beverly, Massachusetts

A.G. Hdr. July 21, 1943

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DRYING SYSTEM (excluding reactivating system)

- 1) Drying Chamber
- 2) Adsorber, with Silica Gel
- 3) Filter
- 4) Circulator for Nitrogen
- 5) Heat Exchanger (water cooled)
- 6) Vacuum Pump and System
- 7) Nitrogen Supply and Gauge Line
- 8) Trays for Charge
- 9) Shelves
- 10) Safety Valve Line

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A.C.H.JR. July 23, 1943

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PLATE V

HIGH FREQUENCY INDUCTION FURNACE

- 1) Quartz Tube and Induction Coil (water cooled)
- 2) Crucible and Cover (graphite)
- 3) Stopper Rod (graphite)
- 4) Mold and Plug (graphite)
- 5) Jacket-Base (water cooled)
- 6) Vacuum, Argon and Helium Line
- 7) Water Inlets and Outlets
- 8) Base (bolted on)
- 9) Cover (water cooled)
- 10) Vacuum Seal and Port for Optical Temperatures
- 11) Weight (removable)
- 12) Capacitor Leads
- 13) Generator Leads

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Beverly - Massachusetts

A.C.H. Jr. July 26, 1943

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