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ATMOIZATION METHOD OF MAKING URANIUM POWDER

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For The Atomic Energy Commission

H. R. Cancell
Chief, Declassification Branch

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ABSTRACT

Atomized uranium powder was produced by forming an electric arc between two uranium electrodes in an inert atmosphere and sending a high velocity stream of inert gas through the arc.

Uranium particles obtained by this method were of spherical shape; smaller particles contained mostly small grains, and larger particles were characterized by larger grains. The particles were ductile and could be hot-pressed to a compact of high density.

The temporary equipment used for these preliminary tests on atomization was not adequate to control particle size. The report, however, includes suggestions for the production of atomized uranium powder of controllable quality.

I. INTRODUCTION

Uranium powder produced in the conventional manner by the decomposition of uranium hydride is characterized by extremely small particle size. Electron microscopic investigation of such powder, revealed a large percentage of particles in the 0.1 to 0.5 micron range. The determination of particle size distribution in this range is, of course, extremely difficult and of questionable accuracy. This small particle size is undesirable, because of the large total surface area that results in rapid oxidation of the powder as well as of the compacts pressed therefrom. In addition, conventional pressing and sintering of compacts made from this powder yields a material of fairly low density, and no means have been found for controlling the porosity of the sintered compact. Tests aimed at variation in particle size of the uranium hydride or the decomposed hydride indicated that it was impossible to obtain larger particles.

Since the atomization method seems to have possibilities of producing a powder of larger and controllable particle size, with a relatively small surface area and therefore less oxidation, preliminary attempts were made to atomize uranium.

II. ATOMIZATION OF URANIUM

Atomization is generally accomplished by forcing molten metal through an orifice and by breaking up the stream of metal by a stream of air, an inert gas, water vapor, a rotating disc, or simply by allowing it to fall into a liquid. Uranium, however, because of its extremely reactive nature is not readily adaptable to any of the above general methods. An attempt was made, therefore, to prepare uranium powder by forming an arc between two uranium electrodes in an inert atmosphere and sending a high velocity stream of inert gas through the arc. Unlike the method of melting where the molten metal is in contact with the crucible, this method of atomization has the advantage of producing small amounts of the metal at elevated temperature which have no other contact than with the inert gas; the method is based on the design principle of some metal spray guns.

In order to study the feasibility of the atomization process for uranium, preliminary experiments were made whereby two uranium electrodes were inserted in a dry box filled with argon (99.6% purity) or helium (99.9% purity). One electrode, a uranium rod one inch in diameter and six inches long was held stationary, and the other uranium electrode (wires varying from 0.09 to 0.25" in

II. ATOMIZATION OF URANIUM (cont'd)

diameter) was moved by hand. The direction of electrode feed was varied; some runs were made with horizontally arranged electrodes and other runs were made with electrodes arranged in a vertical plane. The power source was a motor generator (100 amp. maximum on the d-c side). A high-velocity stream of gas (argon or helium, approximately 375-400 cu.ft./hr.) was sent through a nozzle orifice of approximately $1/32$ " in width and about $3/8$ " in length and was directed into the center of the arc. (It should be noted that the stream of gas covered not only the metal vapor in the arc, but also the molten material on the face of the electrode). This high velocity stream of gas broke the molten and vaporous material into particles and transported them over horizontal distances of approximately 12-15". The particles were collected in a receptacle which was held in the line of particle flow. During the course of several runs, the dew point of the atmosphere and the dry box dropped radically from approximately minus 55-65°C to minus 30-35°C, probably caused by the surface of the walls giving off moisture as the dry box temperature increased from the arc, the temperature of which was estimated at over 3000°C.

It seems that the method of atomization is promising, however, it is obvious that many variables must be controlled in order to obtain the optimum yield of desired powder. Among them are voltage, current, and current density, size and shape of the uranium electrodes, the speed of wire movement, the direction of approach of electrodes (horizontally or vertical), the gas atmosphere, the pressure and velocity of the gas, and the shape and arrangement of the jet.

III. CHARACTERISTICS OF ATOMIZED URANIUM

The uranium particles obtained by this method can be characterized as follows:

1. Most of the particles obtained by this method approached the spherical shape.
2. The particle sizes varied between <44 and 2000 microns, with a great predominance of the large particles.
3. Most of the particles were covered with an oxide film apparently caused by the inadequacies of the equipment used. It was possible to remove the oxide film by cleaning the powder in nitric acid.

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III. CHARACTERISTICS OF ATOMIZED URANIUM (cont'd)

4. The high ductility of this powder was shown by hammering the particles to thin flat discs and by compacting in a die.

The following observations were made on the particles obtained during the preliminary runs:

A. Particle Size and Grain Structure

The typical screen analysis of the powder of one of the runs in argon is shown in Table I.

TABLE I

Mesh Size	Percent by Weight
+10	17.3
-10 +20	55.4
-20 +40	19.5
-40 +60	5.3
-60 +80	1.4
-80 +100	0.4
-100 +140	0.4
-140 +325	0.2
-325	0.1

Particles of the sizes above +40 mesh are too large for powder metallurgy purposes, although they might be useful for other applications. However, experience with other metals, especially copper, prepared by similar methods of atomization, showed that the particle size can be widely controlled by the variables mentioned above especially the current density, the speed of electrodes and the gas flow. Although these variables could not be properly studied with the equipment described above there is every reason to expect finer uranium powder when produced under proper conditions.

III. CHARACTERISTICS OF ATOMIZED URANIUM (cont'd)

Most of the particles were of spherical shape as shown in Figs. 1 and 3. From these figures, and from the metallographic investigations of many other particles, the grain size of the particles appeared to be correlated to a certain extent to the particle size and to the type of gas atmosphere used for breaking the molten metal. In general, but not always, smaller particles contained smaller grains and larger particles contained larger grains. It seems further that particles obtained in argon atmosphere contain larger grains than particles of similar size obtained in an atmosphere of helium. These observations can be explained in the following way - smaller particles cool much faster than large particles. Particles formed and cooled in helium cool much faster than those formed and cooled in argon, on account of the greater heat conductivity of helium compared with that of argon. The cooling time affects the grain size - fast cooling is characterized by fine grains and slow cooling by larger grains, as shown in Figs. 1, 2, and 3. The speed of cooling and the cooling medium also affect the rate of particle oxidation.

B. Purity of Atomized Powder

One of the most important considerations in evaluating the usefulness of atomized uranium powder is the purity of the material. The atomization process practically excludes additions of metallic impurities even at runs made with the imperfect equipment used for these preliminary tests. Spectrographic investigations actually did not reveal any differences in the metallic impurities of the atomized powder compared with the electrodes from which the powder was obtained.

Gas analyses, however, revealed fairly large differences; especially differences in the oxygen content between the atomized powder and the uranium electrodes. It was observed that most of the particles having a black coating could be cleaned by etching with diluted nitric acid and by drying with acetone. The powder particles shows a typical metallic gray surface after cleaning. Table II shows the hydrogen, oxygen and nitrogen content of the cast uranium from which the electrodes were made, of the electrodes and of the powder of several runs before and after acid cleaning. These data show that a large amount

III. CHARACTERISTICS OF ATOMIZED URANIUM (cont'd)

of the oxygen content is on the surface of the particles and can be removed by acid cleaning. An analysis of samples #7 and #8 showed that the oxygen content is a function of the particle size and that finer particles contain relatively more oxygen than coarser particles. In order to evaluate the gas analysis of the particles, one has to consider that the particles were produced in an inadequate and imperfect apparatus and under crude conditions. The high amount of oxygen cannot be attributed to the process or to the gas atmosphere in which the process occurred but rather is caused by the water vapor formed in the dry box during the runs.

TABLE II

Sample No.	Material Tested	% H ₂	% O ₂	% N ₂
1	Cast uranium from which the electrodes are made.	0.003	0.020	0.004
2	Wire electrode 0.09" diameter, clean	0.001	0.026	0.005
3	Run #4 powder, atomized in argon (dew point - 40°C) before cleaning	0.002	0.108	0.013
4	Run #4 powder, after acid cleaning	<0.001	0.070	0.012
5	Run #5 powder, atomized in helium (dew point - 65°C) before cleaning	0.001	0.090	0.009
6	Run #5 powder, after cleaning	0.001	0.031	0.006
7	Run #8 powder, atomized in argon (dew point - 35°C) particle size -60 +80 mesh, after cleaning	0.003	0.125	0.041
8	Run #8 powder, particle size -10 +20 mesh, after cleaning	0.001	0.079	0.009

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III. CHARACTERISTICS OF ATOMIZED URANIUM (cont'd)C. Hardness and Ductility

Further evaluation of the atomized uranium powder was made on the basis of hardness. The hardness of the powder particles is, in general, higher than that of the uranium electrodes from which they were atomized. The Vicker's hardness of the particles atomized in argon was between 365-430 dph, whereas the hardness of the uranium electrode was approximately 240 dph.

Ductility tests were made in two ways: (a) by hammering individual particles and (b) by pressing a few grams in a die similar to that used for the compacting procedure in powder metallurgy. Hammering showed that the spherical particles flatten without cracking, which indicates a fairly high degree of ductility. The particles were pressed in a double-action die of 3/8" diameter at 50 tsi. The pressed density of the compact was 14 g/cc, which is higher than the density of uranium powder made by the hydride method and compacted under similar conditions. This, too, indicates the flattening of the particles during pressing and the high ductility of this powder.

D. Sintering of Atomized Powder

The rate of sintering is a function of the particles and void sizes in the compact and a very low sintering rate could, therefore, be expected with compacts made from the large atomized uranium powder particles. As described above, the powder was pressed at 50 tsi and compacted to a density of 14 g/cc. During sintering in vacuum (0.01 micron) at 1090°C for three hours, the compact expanded and the density decreased to 11.5 g/cc. The decrease in density was probably due to surface oxidation and the low density was to be expected on account of the large pores in the pressed compact and on account of the relatively short sintering time. However, even during this short time, perfect diffusion bonds between the particles, and also bridges between the particles were formed, as shown in Figs. 4 and 5. The sintered compact was substantially stronger than the compact in the as-pressed state. Another specimen pressed in a single action die at 70 tsi had a green density of 14.8 g/cc. Sintering

III. CHARACTERISTICS OF ATOMIZED URANIUM (cont'd)

this compact in vacuum at 1100°C for six hours produced a similar effect as on the above described specimen, i.e., growth of dimensions, and consequent production of density to 13.85 g/cc. The grains of this compact grew during sintering, but to a lesser degree than in similar compacts made from decomposed uranium hydride powder. Sintered compacts of similar low density made from the extremely fine decomposed hydride powder could not be handled in air but burned immediately. The sintered compact made from the coarse atomized uranium particles could be handled in air without any danger of burning.

Hot-pressing the atomized powder in the alpha range (25 tsi at 600°C for 15 minutes) yielded a compact having a density of 18.4 g/cc which indicates the high plasticity of the atomized uranium powder at 600°C (Fig. 6). The hot-pressed compact was practically free of pores. The grains were of medium size similar to the grain size of the original particles. The hot-pressed compact did not have any considerable strength; it seems that the short exposure to a fairly low temperature did not permit the formation of any diffusion bonds.

In the trend of some of our studies concerning the development of compound materials consisting of a metallic matrix and discrete particles of uranium uniformly distributed in the matrix, experiments were made with the spherical atomized powder particles inserted in a solid matrix of fine-grained uranium. For the development of this compound material a small amount of large atomized uranium particles was mixed with the fine uranium powder made by the decomposition of uranium hydride and the mixture was hot pressed in the alpha range at 25 tsi, 550°C for 15 minutes. The density of the hot-pressed uranium mixture was again 18.4 g/cc and the compound material exhibited considerable strength. Fig. 7 shows a structure of this compound material and the complete separation of the particles made by atomization from the matrix made from decomposed uranium hydride. The grain structure of the spherical particles did not change during hot pressing.

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IV. FURTHER DEVELOPMENTS

In order to obtain a controllable product, the process must be done automatically and in an inert atmosphere of high purity. Before designing the pilot plant for the atomization of uranium, further studies are to be made which should include the following:

1. Comparisons of helium and argon as atomizing media regarding their effect on particle size, grain size within the particle, and other properties.
2. The effect of wire diameter and speed of wire movement, voltage variations and various current densities, varying jet pressures, and optimum collection of particles should be determined. These variables should be evaluated on the basis of their influence on particle shape, particle size and particle size distribution, grain structure, stability of grains within the particles at elevated temperatures, oxygen content, hardness, ductility, and compressibility, as well as their sintering characteristics.

The equipment necessary to make the studies should include:

1. A mechanism for moving two uranium wire electrodes which permit the use of various diameters of wire at various speeds of approach.
2. A dry box with optimum dimensions, with tight inlets for wire electrodes, power, etc., and with a pressure release valve to prevent over-pressure.
3. A high pressure gas purification train to be used to purify both the atmosphere in the dry box and the high-speed gas for atomization.
4. A device for collection of the powder which permits fast cooling of the particles thus preventing the formation of agglomerates.

The results described here show that uranium powder can be manufactured by atomization and that modifications of the described method will result in usable particles of controllable sizes.

Any suggestions for the application of the atomized uranium powder for purposes other than powder metallurgy will be welcomed by the authors of this report.

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Fig. 1

Plate 3361

Polarized light 200X

Particle size -40 +80 mesh

Fig. 2

Plate 3360

Polarized light 200X

Particle size +40 mesh

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Plate 3504

Fig. 3

Polarized light 200X

Plate 3368

Fig. 4

Polarized light 200X

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Plate 3364

Fig. 5

Bright Field 200X

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Fig. 6

Plate 3798 Polarized light 200X
 Uranium powder formed by atomization, hot
 pressed at 25 tsi, 600°C, for 15 minutes
 in vacuum.

Fig. 7

Plate 3869 Polarized light 200X
 Uranium powder particles formed by atomization,
 mixed with fine uranium powder made by the
 hydride method, hot pressed at 25 tsi, 550°C
 for 15 minutes in vacuum.

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