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ATOMIC ENERGY DIVISION

W. E. Kingston, General Manager
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MONTHLY TECHNICAL PROGRESS REPORT

June, 1955

Since nontechnical and nonessential prefatory material has been deleted, the first page of the report is page 4.

Contract AT-30-1 GEN-366

Issued: September 13, 1955

AEC RESEARCH AND DEVELOPMENT REPORT

Atomic Energy Division
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I. DEVELOPMENT ENGINEERING "A" (J. L. Zambrow, Manager)

A. Carbon Reduction of Uranium Oxide (R. B. Holden, R. M. Powers and O. Blaber)

Objective: To develop low cost methods of preparing uranium metal from uranium oxide.

Studies are continuing on the preparation of uranium metal by direct reduction of uranium oxide with carbon.

1. Reduction

- a. Thirty-Liter Furnace: The installation and leak testing of the 30-liter furnace for vacuum reduction of uranium oxide with carbon was completed. With a packing of Norblack insulation, the system was pumped down over a three-day period to a pressure of 130 microns. Static leak-testing over a seven-day period showed a pressure rise of about 1.5 mm, corresponding to a leak rate of approximately 0.2 mm/day in a volume of 50 cubic feet. The de-gassed Norblack may be acting as a getter, however, in which case the observed rate may be falsely low.

Attempts to heat an empty crucible in this furnace revealed the need for modification of the power supply, which is inadequate to bring the crucible to red heat. The equipment for this change has been ordered and is promised for delivery in late July.

- b. Three-Liter Furnace: Several reduction runs were made in the three-liter furnace. Two attempts were made to prepare a biscuit in a graphite crucible lined with carbon paste. The paste, a mixture of carbon and tarry pitch, was smeared in place and then baked to produce a liner. In the first run no biscuit was formed, and part of the charge apparently soaked into the liner. In a second attempt, the charge was heated to a final temperature of 2250°C. The biscuit was completely carbided and only UO₂ was obtained after leaching. The liner in each case appeared quite porous and friable though not severely attacked. Nevertheless, this porosity, apparently resulting from loss of the original tar content, makes it unpromising as a protective liner.

Three runs were made using less expensive carbon blacks as the reducing agent, in place of acetylene black currently employed. All three produced satisfactory biscuits in yields of 74 to 78%. The bulk density of the raw charge varied from 2.5 g/cc to 3.2 g/cc, compared with about 2.0 g/cc for charge made with acetylene black. The heaviest of these, Norblack, was submitted for spectrographic analysis to determine its chemical suitability as a reductant. The chief contaminant is silicon. Sponge obtained using this reductant will be analyzed spectrographically.

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I. DEVELOPMENT ENGINEERING "A" (cont'd)

2. Leaching

Experiments are continuing to determine the relative leaching rates and yields of uranium sponge from biscuit subjected to stress by two forms of cold working and by quenching. The biscuits were cold-worked by forging and by pressing.

a. Cold Working

(1) Forging

In order to confirm the decreased leaching time and the 50.7% yield of bright uranium sponge resulting from leaching of a cold-worked biscuit segment, as reported in last month's report, a duplicate segment of the same biscuit was jacketed in steel under argon and forged at room temperature. The resultant segment was still in one piece but exhibited widespread fissuring. Leaching was completed after 43 hours in 40°C-aerated water, containing a detergent. The yield of bright sponge was 50.1%, representing a uranium recovery of 97.5% based on a 51.3% metal content of the biscuit, as estimated from the density of the biscuit (15.9 g/cc).

(2) Pressing

A 40 lb. biscuit was cut into ten sections with densities ranging from 14.48 g/cc (containing 21% uranium) to 16.12 g/cc (54.6% uranium). A poor quality sample, with a density of 14.48 g/cc, was pressed under paraffin oil at approximately 20 tons with hardened steel ball bearings to give intense localized stresses. The segment was crushed into a number of pieces. The oil was displaced with petroleum ether and then with acetone prior to leaching in 40°C-water containing a detergent. Leaching was completed after two days and a 18.9% yield of bright sponge resulted, representing a 90% recovery of metal from the biscuit.

(3) Comparison of Cold Working Methods

A comparison of forging and pressing methods for crushing the biscuit is being made. Samples of biscuit with comparable densities are being cold worked by each method. In these experiments samples are sealed into flattened steel tubes under argon before being worked.

In an experiment still in progress, two samples of biscuit (density = 15.97 g/cc) were employed. The first was pressed under oil at 50 to 60 tsi, to 40% reduction in height. The second was forged at 5000 ft-lbs until reduced approximately 50% in height. After 14 days in 40°C aerated water, contain-

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I. DEVELOPMENT ENGINEERING "A" (cont'd)

ing a detergent, leaching was not complete, but 44.85% bright uranium sponge was obtained from the pressed biscuit. In the case of the forged biscuit, after four days in an identical medium, leaching was not complete; but 44.3% bright uranium sponge was obtained. Anticipated total yields will probably approach 47.5% for the pressed biscuit and 51% for the forged biscuit.

In another experiment in progress, two steel-jacketed samples of 16.12 g/cc density (54% U) were cold worked. One sample was pressed at 62.5 tsi; the other was forged six times at 5000 ft.-lbs per blow. No appreciable difference in leaching rates was found for the first four days of leaching. The total yields of each are expected to be slightly greater than approximately 50%.

These results suggest that forging is more effective than pressing as a means of increasing the leaching rate of the biscuit.

b. Quenching

Quenching was investigated as an alternative to cold working for accelerating the formation of uranium sponge from the uranium-uranium carbide biscuit.

A segment of biscuit with a density of 15.89 g/cc (approximately 51% U metal content) was heated in an argon atmosphere at 850°C and quenched into a large volume of stirred water at 15°C. It was then leached in 40°C-aerated water, containing a detergent. Sixteen days were required to leach the biscuit completely and a yield of 45.6% metal was obtained. Although this yield was slightly better than the 44% yield obtained for an untreated control segment, it fell considerably short of the 43 hour leach time and the 50% yield obtained for a forged segment of the same biscuit. On the basis of this experiment, it appears that quenching is far inferior to cold forging as a means of increasing the leaching rate.

3. Sponge Consolidation

The sponge resulting from the leaching studies is being consolidated by casting or by hot rolling.

a. Casting

Several ingots of uranium have been prepared by induction melting the sponge under vacuum in a thoria-coated zircon crucible and bottom pouring into a water-cooled copper mold. In one melt, the sponge was cold pressed under argon to form a hollow cylinder which was placed in the crucible. The system was

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I. DEVELOPMENT ENGINEERING "A" (cont'd)

evacuated to a pressure of 1 micron and the charge was heated to 1370°C. The pressure in the system was maintained below two microns for most of the heat. The rough density of the ingot (18.82 g/cc) increased to 19.01 g/cc after it was cropped to eliminate a small pipe.

b. Rolling

A quantity of bright vacuum-dried sponge was loaded into a mild-steel jacket under argon and a plug with an evacuation tube was then welded to the open end. The filled jacket was heated to 570°C under a poor vacuum (in excess of 25 microns) and sealed. It was reheated to 600°C and rolled at 10% reduction per pass with reheating between passes. The final thickness of the uranium, after the removal of cladding, was 0.0334", its width 2.94" and its length 15.4". The density of the strip was 18.92 g/cc.

B. Uranium Slug and Slug Canning Development

Objective: To develop a low-cost method of producing dimensionally stable slugs having randomly oriented, ultrafine grain structure; to improve hot-pressing methods for producing slugs of four or more inches in length; to improve canning methods; to develop slug test procedures.

1. Hollow Fuel Elements (H. M. McCullough, R. E. King, J. Fugardi and C. Romero)

Methods have been studied to fabricate hollow fuel elements eight inches in length by hot-pressing uranium powders. This hollow slug design is being considered for purposes of cooling the slug during operation in reactors at higher power levels than are possible with solid cylindrical elements of the same size. A low-cost powder metallurgical method is now being developed.

In previous attempts 1/4" diameter untapered core rods made from 18-4-1 tool steel could not be ejected from an eight-inch long hollow element. Several attempts were made to eject a similar core rod made from Inconel-X without success. Inconel-X core rods 3/8" and 3/4" in diameter, without tapers, could be ejected without difficulty.

The lower limiting diameter for untapered core rods for an eight-inch long element appears to be 3/8"; however, over the same length a 1/4" diameter core rod tapered 0.015" per foot can be removed readily.

I. DEVELOPMENT ENGINEERING "A" (cont'd)

2. Porous Elements (H. M. McCullough, J. R. Bedell, and R. B. Wagner)

Methods are being studied to fabricate porous type fuel elements which are being considered for use in power reactors where long life fuels are required. Any dimensional instability of the uranium would presumably fill the pores instead of distorting the external shape of the element. In the fabrication of porous elements, the two aims currently being pursued are the fabrication of an element with a uniform porosity along the length of the element but not necessarily across its diameter, and the fabrication of an element with a high-density shell and a porous core.

The study on the effect of uranium particle size distribution upon properties of hot-pressed samples is continuing. It has been found that as the powder particle size decreases, the differential between continuous porosity and overall porosity decreases, except for the -325 mesh fraction. For example, the -12 +80 mesh fraction hot-pressed to a density of 16.8 g/cc gave an overall porosity of 11.4% and a continuous porosity to the surface of 3.2%, and the -230 +325 mesh fraction hot-pressed to the same density and overall porosity had a continuous porosity of 9.3%. However, the -325 mesh fraction hot-pressed to the same density and overall porosity had a continuous porosity of 6.9%.

A high-density powder which was essentially -12 +80 mesh and with a tap density of 11.5 g/cc was hot-pressed to the same density conditions. This compact had a continuous porosity of 7.8%.

In transverse rupture tests (1.75" span and a 3/8" diameter bar) the strength decreased with decreasing particle size. The -12 +80 mesh and -325 mesh fractions gave modulus of rupture values of 60,000 and 45,000 psi, respectively. The high density powder, though, gave values of only 20,000 psi. It is thought that the severe cold-working to obtain the high density powder did not permit the plastic flow necessary during hot-pressing to effect good sintering.

C. Preparation and Handling of Fine Non-Pyrophoric Uranium Powder
(S. Storchheim)

Objective: Study of producing and handling non-pyrophoric uranium powder and the basic principles established so that production and handling on a larger scale can be started.

Previous studies here have shown that uranium densities of 0.04 to 0.07 g/cc below that of vacuum hot-pressed standards are attainable by air-hot-pressing techniques. Xylene was found to give higher densities than paraffin oil for non-pyrophoric powder. Intensive study of the preforming operation showed that laminated surfaces on the machined slugs could be eliminated by proper techniques in applying the aquadag lubri-

I. DEVELOPMENT ENGINEERING "A" (cont'd)

cant to the preforming dies. The best preforming pressure was established as 30 tsi and it was found that the results were reproducible. All slugs had excellent surfaces after centerless grinding 0.015" from their diameters. Studies of the physical properties of the slugs showed that hardness is comparable with vacuum-pressed standards and transverse rupture strength is a direct function of the final slug density for both air- and vacuum-hot-pressed slugs.

This month further work was done on preforming lubrication techniques. This work showed that the standard aquadag technique is superior to zinc stearate and stearic acid lubricants for non-pyrophoric uranium. The following results were obtained:

1. Two preforms were made for each lubricant, zinc stearate and stearic acid, with die insert surfaces in the oxidized and non-oxidized condition. Every cold-pressing gave loud "clicking" noises throughout indicative of "hang-up" of powder.
2. As a control, the standard aquadag method was used immediately following the above runs. This resulted in little or no "hang-up" in cold pressing.

In relation to the hot-pressing cycle, a study of the effect of pressure and time at pressure at 600°C was carried out to determine the resultant characteristics of slugs. It was found that a pressure of 12 tsi held for 6 to 15 minutes had very little effect on the resultant densities of the slugs. Further work is being done on this aspect of the problem to establish the best possible procedure for quality and economy in production.

Some preliminary experiments were performed in relation to characteristics of slurries. The following preliminary results were obtained:

1. It was found impractical to determine flow rates since settling of powder in xylene is almost instantaneous.
2. Volumetric measurements on two batches of powder showed a 6% variation in absorption; i.e., 2000 gms. of powder will absorb from 375 to 400 cc of xylene. It is believed that some of this variation is inherent in the technique used and further work is required on this phase.

In view of the large quantities of uranium powder in slurry form required for mixing studies, the safety department recommended a new ventilation installation for the Lee Mixer. When this is completed work will proceed on mixing studies.

Preparations were made for canning a number of air- hot-pressed and vacuum- hot-pressed slugs. This work is in progress and will be reported on in the future.

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I. DEVELOPMENT ENGINEERING "A" (cont'd)D. Separation of Alloys

Objective: To evaluate methods of separating various alloying metals and metallic fission products from fuels and fuel elements.

2. Magnetic Separation (S. Storchheim and I. Scheinhartz)

Magnetic separation techniques depend on the fact that uranium hydride is reported to be magnetic below -100°C . Accordingly some exploratory work was reported in the last monthly progress report using permanent magnets and vibrating equipment to separate particles of uranium hydride from zirconium hydride. The hydrides were contained in a glass tube immersed in a liquid nitrogen bath. Results showed that in magnetically separating a hydrided 4% uranium- Zircaloy 2 synthetic alloy both field strength and method of vibration had an important effect on the efficiency of the method.

In order to determine whether the uranium in an STR fuel element could be converted to a ferromagnetic hydride, a special test was run. A comparison of hydrided synthetic mixes and hydrided STR fuel element samples was made by weighing samples in special electromagnetic equipment. The following conditions were used.

1. No magnetic field, at room temperature.
2. Magnetic field, at room temperature.
3. Magnetic field, at liquid nitrogen temperature.

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I. DEVELOPMENT ENGINEERING "A" (cont'd)

Results showed that the hydrided STR fuel element contained more iron than the hydrided synthetic mix. However, the amount of what is believed to be ferromagnetic uranium hydride contained in each sample was approximately the same.

The magnetic method was also tried previously on a synthetic mix of hydrided uranium - uranium carbide and results were encouraging. This month a similar experiment was performed on a hydrided sample of uranium - uranium carbide biscuit material. The carbon content was reduced from 2.1% to 0.7% in this procedure.

At the present time new equipment is being developed to eliminate the crudeness and increase the efficiency of the method. A gauss meter has been obtained for establishing quantitative data in future experiments.

E. Reduction of Thorium Oxide (R. B. Holden and N. Fuhrman)

Objective: To investigate methods for the production of low-cost thorium.

1. Calcium Reduction

Studies are continuing on the calcium reduction of thorium oxide. Four reduction runs were made in which the charge of ThO_2 , CaCl_2 and calcium was heated to 1080°C in a molybdenum-lined Inconel reactor. The thorium raw material was pre-fired to reduce its carbon content. Redistilled calcium metal was used. The yield of coarse thorium powder was 83% for Run 33, 88% for Run 34 and 86% for Runs 35 and 36. Chemical analysis of the thorium powder indicates that there has been very little change in powder quality.

Two 30 gm samples from each of the four runs were arc melted into buttons. The density of the buttons averaged 11.68 g/cc and the hardnesses are all under Rockwell F50, reflecting the low carbon content of the thorium powder.

Work was continued on the break-out problem in search of a suitable liner material from which the reduction cake can be removed easily in a form ready for crushing. Preliminary experiments with graphite boats in a horizontal tube furnace indicate that graphite may be such a material if the surface-to-volume ratio of the vessel is made low enough.

2. Consumable Electrode Arc Melting of Thorium (S. Storchheim and R. Witt)

Studies were continued on developing an arc melting technique to obtain small ingots from calcium reduced thorium powder. After the best possible ingots are obtained they will be evaluated for physical, chemical and deformation characteristics.

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I. DEVELOPMENT ENGINEERING "A" (cont'd)

Preliminary work reported on last month showed that the higher the power ($P = I^2R$) used, the better was the quality of the resultant ingot. However it was found that higher power inputs led to difficulties such as sticking of ingots to the crucible wall during attempts to eject the ingot. It was concluded that the equipment must be modified to provide better manual arc feed and better cooling of the copper crucible to eliminate this effect. This work also showed that at higher power inputs a ratio of 8:3 or above for crucible diameter to electrode diameter is required or arcing through the crucible occurs.

This month the following modifications were made in the arc melting equipment:

1. The electrode was suspended from a spring to provide better manual feed control.
2. The flow of water around the copper crucible was changed to increase the velocity of water on the outer surface of the crucible. To accomplish this a 3/4" water line was installed and the volume of the water jacket was reduced.

As a result of the above modifications, arcing-through of the crucible walls was eliminated for high amperages as well as sticking of the ingots in the crucible on ejection. Good ingots were made using 600, 700 and 800 amperes at the arc. In all these runs no sticking of the ingot to crucible was encountered. Splatter was also kept to a minimum. The ingot made using 700 amperes had a 3/16" pipe at the top. This effect was eliminated in the 800 ampere ingot by reducing the current to 400 amperes and "puddling" the melt at the end of the cycle. All these ingots were made in an argon atmosphere at a pressure of 23 inches of Hg. These ingots are being evaluated for physical and chemical characteristics which will be reported in the future.

One experimental melt was tried in vacuo at 40 μ . The result was a glow discharge above 250 amperes and the run had to be stopped.

F. Preparation of Beryllium (R. B. Holden and M. C. Kells)

Objective: To produce low-cost beryllium chloride; to evaluate the possibility of producing low-cost beryllium metal by electrolysis of beryllium chloride into a mercury cathode with a low-melting electrolyte.

1. Chlorination

The preparation of beryllium chloride by the chlorination of beryllium oxide is being investigated.

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I. DEVELOPMENT ENGINEERING "A" (cont'd)

Analytical data for Run 10, described in last month's report, indicated a chlorine efficiency of approximately 93% and a greater than 90% conversion of BeO to BeCl₂. The latter figure is an estimate since scale from the reactor wall was mixed with residual charge so that the exact conversion could not be determined.

Beryl ore ground to -325 mesh was analyzed for water content by heating at 1000°C for about 30 minutes and then heating at 1200°C for about 30 minutes. The water removed is as follows (reported as percent of original weight of beryl).

at 1000°C 2.35% H₂O
at 1200°C 0.043% additional H₂O

2. Electrolysis

Approximately 335 grams of beryllium amalgam were prepared by the electrolysis of BeCl₂ (prepared from BeO) in the 5-inch stainless steel cell. The cell was operated for 200 ampere-hours with a current efficiency of 50 to 70%. The mercury was removed by vacuum distillation. A sinter cake resulted and a white deposit appeared on the condenser arm. This deposit consisted of BeCl₂ and NaCl. The sinter cake analyzed 2.13% Cl⁻ and 10.4% BeO. A leak in the side arm of the stainless steel cell and attack of the Pyrex distilling flask may account for the high oxygen content. The reason for the high salt content of this amalgam, is not understood.

Preparations were made for putting the 10-inch stainless steel cell into operation. The cell was resistance wound and insulated. Beryllium chloride was purchased and is being analyzed.

II. DEVELOPMENT ENGINEERING "B" (R. S. Stewart, Manager)

A. Savannah River Fuel Elements (W.G. Lidman, N. Grossman and M.H. Binstock)

Objective: To investigate and develop methods, by means of which flat plate fuel elements approximately 14 feet long can be manufactured economically by powder metallurgical processes and to investigate methods for producing tubular fuel elements.

The development of methods for fabrication of long flat fuel elements was continued. During the month, work was confined primarily to product evaluation. Nondestructive evaluation of bonding with ultrasonic pulse-echo techniques showed good bonding, with some areas showing low amplitude sound echos from the core. Metallographic examination of these areas showed the presence of an unidentified black layer between the uranium and the bonding zone. Samples of this material have been analyzed by X-ray diffraction methods, and the black layer has been identified as uranium hydride. Efforts are being made to eliminate or minimize this layer.

In order to evaluate the product, a number of finished plates were cut up for testing. Overall density of the plates determined from measurements at 20 positions on each plate showed variation of from 18.6 to 19.0 g/cc with an average of 18.8 g/cc. Grain size was 4 to 25 microns, with occasional clusters of grains ranging up to 50 microns diameter. Hardness of the core ranged from Rc 23 to Rc 29.

The construction of a suitable autoclave for the testing of 14-foot flat plates was completed. The autoclave operates with 175°C saturated steam. The usual test is of 40 hours duration. Provisions were made in the original design for the simultaneous testing of five fuel elements. After a few trial runs it became apparent that the proximity of the plates may seriously handicap their removal should a failure occur. In the final design three plates can be simultaneously tested.

Investigations were carried out to determine the cause of autoclave failures of flat plates. One tentative theory is that the plates developed leaks between the end plugs and the aluminum sheaths. The end pieces of several flat plates that had failed in the autoclave were leak tested with a helium leak detector; no leaks were revealed. To further test this possibility, weld beads were laid all around the ends of two plates and these two plates were then tested in the autoclave. After two runs, the plates were removed for inspection. One of the welded plates started to fail. The failures started at the ends but the mechanism of the failure is still not apparent.

In an attempt to separate the possible cause of failure due to heat alone and corrosion alone, two flat plates which have not been previously autoclaved have been placed in a 16-foot furnace within a pipe and tested in air at 170°C for 48 hours.

II. DEVELOPMENT ENGINEERING "B" (cont'd)

B. Hydrostatic Pressing of Metal Powders (W. G. Lidman)

Objective: To evaluate mold designs and mold materials for the compacting of powdered materials, such as thorium and uranium, and to evaluate the feasibility of hot hydrostatic pressing.

Preparation of samples was started to compare properties of metal powder compacts pressed hydrostatically with samples pressed in steel dies. Compacting was started of samples of aluminum, copper, brass, nickel, silver and iron powders. Pressures used for compacting were 20, 30, and 40 tsi.

The pressure intensifier which was purchased for use with the 4-inch bore cylinder and for hot hydrostatic pressing was received.

C. Slip Casting of Metal Powders (W. G. Lidman)

Objective: To develop and evaluate methods of slip casting metal powders.

The work on slip casting was concluded during the month of June. A report summarizing the results of the year's work is being prepared. Samples were prepared during the year by slip casting using stainless steel powders. The properties of this material were compared with samples produced by cold-pressing-and-sintering and by conventional methods. The highest density achieved by slip casting was 7.08 g/cc. The best properties, however, were obtained with a sample which had a density of 6.62 g/cc. This sample had an ultimate strength of 70,500 psi with a yield strength of 40,300 psi, and an elongation of 20 per cent. This indicates that slip composition which produces highest density does not necessarily produce the best properties.

III. DEVELOPMENT ENGINEERING "C" (L. W. Kates, Manager)

A. Dimensionally Stable Uranium Alloys (H. S. Kalish and R. P. Wittmann)

Objective: To develop powder-metallurgical methods for preparing uranium-rich alloys which have a fine grain structure and are dimensionally stable; to prepare samples for tests at this and other laboratories.

Several more pin-type 2 w/o zirconium-uranium alloy samples were thermal cycled for comparison with 0.5 w/o silicon, 1 w/o chromium and 1.4 w/o molybdenum-uranium alloys. These samples were all made by powder metallurgy methods. After 500 cycles through the alpha beta transformation temperature it was clear that the 2 w/o zirconium alloy was the least stable of the group. The chromium and silicon alloys showed considerable distortion after cycling. The molybdenum alloys on the other hand showed the usual consistent stability (less than 0.5% deformation per 100 cycles).

Work is being done on the hydrostatic pressing of cylindrical bars as a means of completely eliminating orientation effects which might be introduced during die compacting. With plastisol molds and special techniques it was possible to make successful 1.4 w/o molybdenum alloy compacts at pressures of 30 tsi. After sintering at 1100°C densities ranging from 18.48 to 18.73 gms/cc (98.40 and 99.73% of theoretical) were obtained. These results indicate that high-density alloy material can be made by this method. Plastisol molds are not completely satisfactory, however, and it is planned to investigate a more flexible mold material such as surgical rubber tubing.

A new type vacuum furnace for sintering uranium alloys was completed. This furnace consists basically of a 6" I.D. Inconel pot which is suspended over a hot resistance wound furnace during the evacuation period. When it is desired to heat the Inconel pot the furnace is moved up so that it surrounds the Inconel pot. This method permits the sintering of a relatively large number of samples in the shortest possible time. Prove-in runs with the equipment indicated that it can be operated up to temperatures of 1175°C and that at temperature vacua on the order of 0.1 micron or better can be obtained. Preliminary alloy samples which were sintered in this furnace were of high density and showed no signs of oxidation. Investigations are now being made to determine the minimum pump down time and the maximum permissible pressure for successful sintering of uranium alloys.

B. Wire Fuel Elements (H. P. Kling and S. S. Shapiro)

Objective: To develop procedure for preparing and evaluating wire type fuel elements.

Six Zircaloy-2 clad 21% U-Zr cored assemblies were fabricated to .060" wire for ANL. They were cut into 36 three-foot lengths and radiographed. Samples were taken from each end for metallography, and all the lengths were sent to the Chemistry Laboratory at Linden Place to remove the molybdenum disulfide lubricant coating. The wire appears to be sound and after cleaning will be shipped to ANL to complete the order.

III. DEVELOPMENT ENGINEERING "C" (cont'd)

C. Perforated Wafer Fuel Elements (H. S. Kalish and E. N. Mazza)

Objective: To develop a fuel element in the form of stacked perforated wafers with tubing or other corrosion-resistant coating inside the perforated wafer holes to carry the heat transfer fluid. The wafers are to consist of uranium oxide in an appropriate matrix or a uranium alloy, and the tubing is to be some compatible material for high-temperature operation.

Progress was made in the investigation of the gas permeability of stainless steel UO_2 core material. Additional attempts were made to improve the density of the 30 w/o UO_2 stainless steel core material.

UO_2 was prepared by firing as received UO_2 for three hours at $1600^\circ C$ in hydrogen. After firing, the UO_2 was screened to obtain the -140 +325 and -325 mesh fractions. An additional batch of UO_2 was prepared by the granulation process developed by GE/KAPL. This method consists of adding calcium fluoride, Cerumel "C" and water to the UO_2 , milling, drying, pelletizing, grinding, firing and regrinding. The granulation technique results in a yield of approximately 56% of -140 +325 mesh UO_2 as compared to a yield of only 20% -140 +325 mesh UO_2 by the ordinary firing method.

For matrix material both -200 and -325 mesh stainless steel of type 302B and a special high silicon type 347 stainless steel was used. Small cylinders were compacted at 50 tsi from all combinations of UO_2 and stainless steel described above. Compacts were sintered for 16 hours at $1350^\circ C$ in dry hydrogen.

Densities varied from 86 to 92% of theoretical, depending upon the materials used. The -140 +325 mesh UO_2 resulted in the highest density along with the -325 mesh stainless steel. Method of UO_2 preparation appeared to have no effect on the density other than the differences in yield described above. All samples were found to be permeable, but a tenfold decrease in permeability was encountered as theoretical density increased from 86 to 92%. Attempts are being made to increase the density of this core material further so that the minimum density at which nonpermeable core material can be obtained is determined.

Molybdenum-30 w/o UO_2 wafers were compacted and sintered for six hours at $2000^\circ C$ in hydrogen utilizing an induction furnace. No improvement in density was obtained over the 85% of theoretical for wafers previously sintered for 16 hours at $1350^\circ C$. A check is being made of the induction furnace to determine the accuracy of the sintering temperature and further attempts will be made to increase the density of this material by high-temperature sintering.

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III. DEVELOPMENT ENGINEERING "C" (cont'd)

D. Mechanism of Dimensional Instability of U Under Irradiation (L. Seigle and R. Resnick)

Objective: To investigate the mechanism of dimensional instability of alpha-uranium.

Efforts to measure the self-diffusion coefficients of uranium are continuing. The technique being employed was described in last month's report. A modification has been made in the cathodic sputtering apparatus, i.e., the addition of a liquid nitrogen cold trap which serves the two-fold purpose of improving the vacuum and preventing the passage of toxic uranium particles into the laboratory's atmosphere. A complete trial run is being performed with natural uranium substituted for the U^{233} which will be used in the final experiment, since such a run is necessary for the preparation of a feasibility report.

Studies of pore formation in gamma brass revealed no unusual features due to anisotropy of vacancy diffusion. Since the porosity formation was apparently normal, no further experiments with this material are planned.

A new series of experiments have evolved from some recent observations of the unusual porosity seen in silver-gold sandwich type diffusion couples. Apparently this is related to vacancy condensation on impurities. The critical impurity is thought to be silver oxide which forms during the diffusion of gold into silver. In order to confirm this opinion, some of the silver used in these couples has been vacuum-melted to remove any oxygen present in solution. This material will now be bonded to gold and annealed in vacuum.

E. Fundamentals of Sintering (L. L. Seigle and A. L. Pranatis)

Objective: To study the fundamental mechanisms of sintering in metals and oxides. Immediate areas of study include an investigation of mass transport during sintering with the aid of fiduciary markers and determination of oxides.

Because of failure of both the vacuum system and the vacuum gauge, work on the sintering of Cu_2O was delayed during the past month. The pumping system has now been cleaned up and repaired and resumption of the dilatometric measurements of the sintering of Cu_2O is awaiting repair of the Alpatron vacuum gauge.

In the interim, work has been resumed on the sintering of marked wire compacts. A series of wire compacts are being prepared with the tungsten markers buried at various distances beneath the surface. No quantitative measurements have yet been made, but metallographic examination seems to indicate that the markers remain circular in cross-section during sintering, thus bearing out further the diffusion theory of sintering in metals.

III. DEVELOPMENT ENGINEERING "C" (cont'd)

F. Fundamentals of Diffusional Bonding (L. Seigle and L.S. Castleman)

Objective: To study the mechanism of pressure welding through a study of the diffusional processes occurring during the formation of metallic bonds and of the effects of pressure on diffusion and upon phase formation.

The bonding apparatus for the study of diffusional bonding is now completed, and the loading mechanism calibrated. It has been determined that loads reproducible to $\pm 2\%$ can be applied to the specimen by loading weights on the lever arm of the arbor press; however, great care must be exercised to minimize frictional effects by vibrating portions of the apparatus immediately after the load is applied and to maintain the lever arm height constant. In order to make loading more foolproof, it has been decided to place two standard compression specimens in series with the specimen during all runs - one above and one below the specimen; strain gages attached to the specimens will enable one to read the applied load at any time during a run.

Three diffusion bonding runs were made at 500°C under a pressure of approximately 5 tsi for 15 minutes, one hour, and three hours; in the latter two cases, tungsten powder was used as a marker material. When the specimens were polished and etched, it was found that the hard tungsten particles had scored the polished surface badly, and in some cases had gouged out portions of the intermetallic layers. In future runs, molybdenum wires or sheet will be used for marker material. The thicknesses of the layers found are currently being measured.

A number of aluminum samples are being electroplated with nickel; as soon as the electroplated samples are available, diffusion runs will be made at atmospheric pressure.

The study of the growth of diffusion layers in our original Ni-Al samples has been concluded. A number of samples which had been bonded under pressures of 5 and 11 tsi for two minutes in the temperature range 525° to 575°C , were given additional diffusion anneals at atmospheric pressure and at 600°C for the following times: 1-3/4, 4, 7-3/4, 20, 45, 66, and 317 hours. When the logarithm of the total layer thickness was plotted as a function of the logarithm of the annealing time, it was observed that the points lay on a parabolically shaped curve; the slope of the best straight line which could be drawn based on a least square analysis turned out to be 2.52. Thus, the growth of the layers in the above case did not seem to take place in the manner which would be predicted by classical solutions of the diffusion equation.

A preliminary study of the intermetallic layers formed during diffusion bonding has been completed. As was reported last month, the phase at the surface of the nickel rich layer was identified as Ni_2Al_3 . This phase persisted below the surface to such a depth that it appears that the major portion of the nickel rich phase which is seen under the microscope is Ni_2Al_3 . Subsequent, more careful x-ray diffraction analyses will be performed on diffusion bonded specimens made of higher purity

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III. DEVELOPMENT ENGINEERING "C" (cont'd)

materials in order to determine if other nickel-rich intermetallic layers exist in appreciable thickness between the Ni_2Al_3 and the nickel-rich terminal solid solution.

A few melting experiments have been carried out with commercial purity aluminum and nickel in a high frequency induction unit under vacuum to determine if the intermetallic compounds can be prepared easily. Trouble was experienced with segregation in the preparation of the melts. If incremental diffusion couples must be prepared for future work, some development work will have to be carried out to perfect the melting techniques required to prepare the samples.

Staff Coordination of Work Under Contract AT-30-1 GEN-366

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