UNCLASSIFIED

~~CONFIDENTIAL~~

SEP-199(Del.)

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

~~RESTRICTED DATA~~

This document contains restricted data as defined in the Atomic Energy Act of 1954. Its transmittal or the disclosure of its contents in any manner to an unauthorized person is prohibited.

This document contains Confidential-Restricted Data relating to civilian applications of atomic energy

~~CONFIDENTIAL~~

UNCLASSIFIED

UNITED STATES ATOMIC ENERGY COMMISSION

MONTHLY TECHNICAL PROGRESS REPORT FOR MAY 1955

Photostat Charge \$ 6.30
Microcard Charge \$.75
for Access Permittees

Available from
Technical Information Service Extension
P. O. Box 1001, Oak Ridge, Tennessee

August 5, 1955

Sylvania Electric Products, Inc.
Atomic Energy Division
Bayside, New York

Technical Information Service Extension, Oak Ridge, Tenn.

AEC RESEARCH AND DEVELOPMENT REPORT

Classification cancelled or changed to UNCLASSIFIED
by authority of 3-30-60
by B Date 4-2-60

DECLASSIFIED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

SEP-199-P

TABLE OF CONTENTS

Page No.

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

A. Carbon Reduction of Uranium Oxide.....	4
1. Reduction.....	4
2. Leaching.....	4
3. Pickling.....	5
B. Uranium Slug and Slug Canning Development.....	5
1. Hollow Fuel Elements.....	5
2. Porous Elements.....	6
C. Preparation and Handling of Fine Non-Pyrophoric Uranium Powder.....	7
D. Separation of Alloys.....	8
1. Uranium Dioxide and Nichrome.....	8
2. Magnetic Separation.....	9
E. Reduction of Thorium Oxide.....	9
1. Calcium Reduction.....	9
2. Consumable-Electrode Arc Melting of Thorium.....	10
F. Preparation of Beryllium Chloride.....	11
G. Foil Elements.....	12

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

A. Savannah River Fuel Elements.....	14
B. MTR Fuel Elements.....	14
C. Hydrostatic Pressing of Metal Powders.....	14
D. Non-Destructive Testing.....	15
E. Product Evaluation of Flat Plates.....	15

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

F. Fundamentals of Sintering.....	20
G. Fundamentals of Diffusional Bonding.....	21

DECLASSIFIED

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

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

Thirty-Liter Furnace: The 30-liter induction-heated furnace for vacuum-reducing uranium oxide with carbon has been received and installed. The vacuum and water leads have been connected and the entire system is being leak-tested. It is expected that this furnace will be in operation by the end of June.

Twelve-Liter Furnace: One reduction run was made in the 12-liter induction-heated furnace prior to dismantling to make room for the newly received 30-liter furnace. By means of pre-firing, a double charge of UO_2 -carbon pellets weighing 113.5 pounds was loaded into the crucible. The reduction was carried out in the usual manner with a pressure of 0.5 atmosphere maintained during the first half of the run. A well-fused biscuit weighing 42 pounds was obtained, which corresponds to a 45% biscuit yield. More than half of the charge was unfused crucible hang-up, probably due to the large temperature difference between the top and the bottom of the crucible. More nearly isothermal conditions are necessary for good biscuit yields.

2. Leaching

A number of carefully controlled leaching experiments has been completed and the results are presented in Table I. In these studies a typical 40-pound biscuit was cut into 16 pie-shaped sections, each weighing about two pounds. The leaching experiments were performed at four temperatures and in three different media.

Confirming the leaching tests last month on smaller biscuit specimens, these results show that the bright sponge yield decreases with increasing leaching temperatures. The study indicates that improved yields are obtained when air is bubbled through the leaching bath; however, more data are needed to confirm this.

A biscuit segment was cracked with a drop hammer. The resultant segment was broken into two large pieces, each of which exhibited a number of small cracks. Leaching of these pieces was extremely rapid; it was largely completed after 24 hours at 40°C, indicating a ten-fold increase in the leaching rate. Preliminary results suggest that the yield will be higher than with uncrushed biscuit.

DECLASSIFIED

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

Experiments on the effect of quenching the biscuit (as a substitute for crushing) are in progress.

TABLE I
LARGE BISCUIT LEACH

<u>Medium</u>	<u>Temp., °C</u>	<u>Yield % (Bright Sponges)</u>
Stagnant H ₂ O	40	42
H ₂ O + Air	40	44
H ₂ O + Air + Detergent	40	44
Stagnant H ₂ O	50	40
H ₂ O + Air	50	40
H ₂ O + Air + Detergent	50	42
Stagnant H ₂ O	60	33
H ₂ O + Air	60	36
H ₂ O + Air + Detergent	60	36
Stagnant H ₂ O	70	30
H ₂ O + Air	70	30
H ₂ O + Air + Detergent	70	31

3. Pickling

It has been found that 0.1N ceric sulfate removes the oxide film from sponge much more rapidly than the standard 1:4 HNO₃ treatment. Its rate of attack on metal appears to be negligible.

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 are being studied to fabricate hollow fuel elements of 1-3/8" O.D. and 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.

DECLASSIFIED

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

The difficulty of ejection of tapered core rods from hollow elements has essentially been overcome. Ejection of the core rod by induction heating the ejected slug containing the core rod and simultaneously applying a load to the core rod has proved to be quite effective.

In the past month, 3/4", 3/8" and 1/4" diameter core rods without tapers were tried in hollow elements eight inches long. In the few runs tried, the 1/4" diameter core rod could not be ejected; but, the 3/8" and 3/4" diameter core rods were ejected without difficulty. The 1/4" diameter rod, however, was made of 18-4-1 hardened tool steel; whereas the 3/8" and 3/4" rods were Inconel-X. The thermal coefficient of expansion is more favorable for Inconel-X which may account for the better results.

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.

In the fabrication of porous elements with a high-density skin and porous core by hot rolling a hot-pressed hollow element in which the core was loaded either with powder or a separately pressed porous core, a considerable amount of preferred orientation resulted in the high-density skin, particularly in reductions greater than 30 percent. Also, no bonding was effected between the skin and porous core.

An attempt was made to achieve a metallurgical bond between a porous core and hollow rod by partially hydriding the two mating surfaces and then hot pressing the assembly at 600°C in vacuum. No bond was achieved and it was concluded that the pressures needed to effect a bond would change or eliminate the porosity in the core.

A study has been initiated to determine the effect of uranium particle size distribution upon the porosity of samples hot-pressed to a given density. It has been found that as the powder particle size decreases the differential between continuous porosity and overall porosity decreases. For example, -12 +80 mesh powder hot-pressed to a density of 16.8 g/cc yielded an overall porosity of 11.4 percent and a continuous porosity (interconnected pores) of only 3.2%; whereas -230 +325 mesh powder hot-pressed to a density of 16.6 g/cc yielded an overall porosity of 11.6 percent but a continuous porosity of 9.3%.

0371228.1030

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

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

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

As shown in last month's report, SEP-198-P, the density difference between a vacuum-hot-pressed standard and a slug air-hot-pressed from non-pyrophoric powder is primarily due to carbon. The carbon content is believed to be partially due to entrapment during the hot-pressing operation and partially due to pyrolysis of the oil during the heating cycle. The density difference noted above only amounted to 0.04 to 0.07 g/cc when xylene was used to coat the uranium particles. However, the surfaces of all slugs had laminated surface structures even after machining the surfaces. These poor surfaces were attributed to use of an excess of aquadag lubricant during the preforming operation. The surface condition was found to have no effect on the density.

As a result of the above findings, an intensive study of the preforming operation was started this month. The objective was to eliminate the poor surfaces and still maintain acceptable densities. In all the work it was decided to use xylene exclusively for coating the uranium particles, because higher densities had been obtained with xylene than with paraffin oil. The results have been positive and the following is a summary of the observations made:

1. Ordinarily the preforming die inserts are lubricated with a heavy coating of aquadag, this is followed by cleaning on a wire wheel, and then the inserts are given a final coating of aquadag. It was found that a light cleaning on the wire wheel leaves a minimum of aquadag on the surface, sufficient to prevent "hang-up". The surfaces on slugs made from preforms pressed in this manner were found to be acceptable.
2. A study of the effect of preforming pressures on final slug density and surface appearance showed that a preforming pressure of 30 tsi was the best. The slug air-hot-pressed from this preform had an acceptable surface after centerless grinding only 0.020" from its diameter.
3. A series of six slugs were air-hot-pressed from preforms made using conditions stated in "1" and "2" above. All these slugs exhibited excellent surfaces after centerless grinding 0.015" from the diameters. Pickling these slugs showed that two of them cracked when dropped in the hot-pressing die, but this can be prevented in the future. The other slugs had acceptable surfaces after pickling.
4. The density of the slugs in this series was 0.04 to 0.07 g/cc below that of the vacuum-hot pressed standard.

DECLASSIFIED

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

Last month it was found that hardness of slugs made from non-pyrophoric powder was very uniform and comparable with vacuum-hot-pressed standards. This month it was established that transverse rupture strength is a function of the final slug density for both air-hot-pressed and vacuum-hot-pressed slugs. The following observations were made.

1. Three vacuum-hot-pressed slugs had rupture strengths ranging from 146,000 to 171,000 psi. Their densities ranged from 18.96 g/cc to 18.97 g/cc.
2. Four air-hot-pressed slugs with densities ranging from 18.77 to 18.91 g/cc had rupture strengths of 29,900 psi to 99,500 psi.

Future work will be concerned with the following:

1. The effects of air-hot-pressing variables, such as pressure and time at pressure, on the final properties of hot-pressed slugs.
2. Mixing and pouring variables of slurries.
3. Canning of hot-pressed slugs.

D. Separation of Alloys

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

1. Uranium Dioxide and Nichrome (R. B. Holden, S. Ruby, C. I. Whitman and V. B. Compton)

Experiments to determine uranium recovery from synthetic mixtures of uranium and Nichrome V dissolved in CuCl_2 have continued. Sixteen experiments, under a variety of conditions, have been made. Recovery has ranged from 80 to 99% with eleven giving recoveries above 90%.

In these experiments Nichrome V is dissolved in CuCl_2 solution containing HCl. A known quantity of uranium is added to the solution. The uranium is precipitated with ammonia, and excess ammonia is added to form the soluble copper, nickel, and chromium complex ammonia ions. The solution is filtered and the precipitate is washed with concentrated ammonia to remove traces of copper and nickel. Iron present as an impurity and some of the chromium remain with the uranium precipitate. For analytical purposes the chromium is removed by oxidation with hydrogen peroxide to the soluble chromate state.

Samples of Nichrome - UO_2 sheet have been received from the General Electric Company plant at Cincinnati. These will be used in evaluating the above method of recovery.

037122A.0330

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

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

An exploratory investigation was begun to determine the feasibility of trying to separate samples of hydrided 4% uranium-zirconium alloy using magnets. The basis of this investigation is the fact that uranium hydride is reported in the literature to be magnetic below -100°C . It was decided to try it using permanent magnets and vibrating equipment to separate the particles while contained in a glass tube immersed in a liquid nitrogen bath. The following results indicated that the field strength had an important effect on the efficiency of the method:

1. With a permanent magnet, the yield of uranium-rich material was low, but chemical analysis showed the uranium content to be 90% of the total yield.
2. The use of a powerful electromagnet resulted in a much larger yield, but there was no uranium enrichment.
3. The method of vibration was also found to be important.

The above method was also applied to the separation of uranium hydride from uranium carbide and the results were encouraging. Starting with a sample containing 2.5% carbon as uranium carbide, the carbon content was reduced to 0.59% by this method. However, the equipment used was very crude and it is felt that the method can be improved. As a result, equipment is now being designed to obtain more complete separation of the carbide.

E. Reduction of Thorium Oxide

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

1. Calcium Reduction (R. B. Holden and N. Fuhrman)

Studies are continuing on the calcium reduction of thorium oxide. Three reduction runs were made in which the charge of ThO_2 , CaCl_2 , and calcium was heated to 950°C in an Inconel reactor. The thorium raw material was pre-fired to reduce its carbon content and handled in an argon atmosphere prior to reduction. Redistilled calcium metal was used. The yield of coarse thorium powder was 85% for Run 30, 80% for Run 31 and 87% for Run 32. Except for a further reduction in carbon content to 0.018%, there has been no significant change in powder quality. A study is being undertaken to determine the optimum conditions for reduction.

Two 30-gm samples from each of the three runs were arc melted into buttons. The density of the buttons averaged 11.69 g/cc and the hardnesses are all under Rockwell F50, reflecting the low carbon content of the thorium powder.

DECLASSIFIED

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

A study has been made of the kinetics of adsorption of carbon dioxide by ThO_2 as a function of firing temperature. The experimental procedure consisted of the following steps: (1) firing the as-received ThO_2 at elevated temperatures; (2) cooling and handling the fired oxide under argon; (3) exposing samples of fired oxide to an air stream for varying lengths of time; (4) analyzing the exposed and unexposed samples for carbon. The results of this investigation are presented in Table II.

TABLE II
ADSORPTION OF CO_2 BY FIRED ThO_2

Firing Temp. °C	Carbon Content (ppm)				
	Unexposed	Exposure Time to Air Stream (Min.)			
		15	30	60	180
600	210	340	410	400	400
700	80	340	420	400	410
800	70	340	350	390	410
900	60	290	290	280	290
975	80	170	210	190	230

Note: Original carbon content of ThO_2 was 380 ppm.

The problem of breaking out the reduction cake from the reactor in a form suitable for crushing and leaching was investigated. Experiments were performed on a small scale to determine the feasibility of employing liners of the following materials: fused dolomite, fused lime, Aquadag, and molybdenum disulfide. Under actual reduction conditions only the fused lime shows promise of being a suitable material. In the case of Aquadag and MoS_2 , normally non-sticking agents, the reduction cake firmly adhered to the walls of the container which had been coated with these materials.

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

From the work on calcium reduction of thorium oxide to thorium powder, it was found that small arc melted buttons made from the powder had low hardness, 38-42 R/F, and high densities, 11.67 - 11.70 g/cc. Work is now in progress to develop an arc melting technique to obtain small ingots from calcium-reduced thorium powder. The ingots will then be evaluated for physical, chemical and deformation characteristics.

According to the literature on arc melting, the consumable-electrode technique with a water-cooled copper crucible is best adapted to refractory metals and alloys. Ordinarily, the melting is done in a vacuum or an inert gas such as argon or helium. Of all the commercial pro-

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

cesses available, consumable-electrode arc melting is stated to yield the purest ingots. The only process that provides higher purity material is zone melting but it is not adaptable for our purposes because of inherent production limitations of this process.

To date several arc-melting runs have been made to get experience in handling the equipment and determine the necessary conditions to obtain good ingots. The following observations have been made so far:

1. The higher the power (i.e., $P = I^2R$, for a thorium resistivity of 30×10^{-6} ohm-cm) used, the better is the quality of the ingot. This relationship was deduced from the following:
 - a. Five runs were made with 13.5 watts to 130 watts (based on resistance of electrode) in various steps. The quality of ingots at the higher arc power inputs had the best visual appearance and were sounder.
 - b. A section cut from the ingot made using 130 watts power had a density of 11.60 g/cc. The hardness was about 85 R_F, but this ingot was made from derby thorium. Ingots made from calcium reduced thorium should have lower hardnesses.
 - c. According to the literature on consumable-electrode arc melting of thorium, 300 to 350 watts was used to obtain ingots 1-1/2" to 7" in diameter.
2. Modification of the existing equipment is necessary to provide better control of manual arc feed and better cooling of the copper crucible so that melting can be tried at higher power inputs. This was deduced from the following observations:
 - a. Sticking of the electrode during a melting run has been encountered during attempts to control the arc feed manually.
 - b. With a power input above 130 watts, leaks occurred in the copper crucible, indicating poor cooling for the quantity of heat generated in melting.

Future work is contemplated on modifying the equipment to obtain more arc power safely to produce the best possible rate of melting.

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

Objective: To prepare low-cost beryllium chloride.

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

DECLASSIFIED

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

Analytical data for chlorination Run 7, described in last month's report, indicated a chlorine efficiency of greater than 99% and a 93.2% conversion of BeO to BeCl₂. Iron content was 0.016%. A chlorine efficiency of 99% and 94.3% conversion to beryllium chloride was obtained for Run 8. Iron content of the product was 0.020%.

Three chlorination runs were completed in the two-inch Vycor tube reactor equipped with a two-liter bulb. In two of these runs chlorine was added above the charge to reoxidize any FeCl₂ to FeCl₃ which passes through the 300°C condenser, thereby eliminating iron from the beryllium chloride. The charge of approximately 125 grams of BeO with a slightly greater than the theoretical amount of carbon was chlorinated at 1000°C, and the beryllium chloride condenser was maintained at about 300°C. In Run 9 the beryllium chloride was quite white in appearance, indicating that the added chlorine produced the desired purification. However, the Vycor joint froze and the BeCl₂ had to be dissolved to free the joint. Preliminary data for Run 10 indicate a greater than 74% conversion to BeCl₂. The iron content was 0.002%. The amount of iron present has been reduced by a factor of 10 by use of additional chlorine above the charge.

In Run 7, described in last month's report, a heavy white scale was observed on the reactor wall, extending from the base of the charge to the condenser bulb. This scale was analyzed and found to be essentially pure BeO. The silica tube showed evidence of heavy attack and a fair quantity of SiCl₄ was deposited in the cold trap during the runs. It appeared that BeCl₂ was attacking the silica and converting it to SiCl₄ while being converted to BeO. To verify this a simple experiment was performed in which BeCl₂ was sublimed through a clean Vycor tube heated to 1000°C for approximately 30 minutes. The Vycor showed obvious attack.

Run 11 was made using an 8 inch long graphite sleeve to contain the charge, in an attempt to minimize the BeO formation on the wall. However, the graphite was too porous and scale did develop. Approximately two-thirds of the total chlorine had passed between the sleeve and wall of the reactor.

Non-porous carbon is being considered as an alternate material for reactor wall construction. Samples of this material are on order.

G. Foil Elements (S. Storchheim)

Objective: Investigate the basic principles of making foil elements by powder metallurgy techniques. This will include the hot-rolling of powders and the sintering of green rolled strips, as well as some studies concerning the metallurgical and mechanical properties of the foils made.

Work was started on the rolling of 18-8 stainless steel foil elements containing cores of 30% and 40% UO₂. Considerable difficulty was encountered at first in preventing the core from coming through the sides

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

of the strip. This was remedied by increasing the lengths of the outer and inner hoppers and also by switching from an inclined slide stop to a horizontal one. As a result the flow started more uniformly. A vibrator on the inner hopper prevented clogging of the restricted opening for UO_2 . Previous experience on the rolling of 18-8 showed that higher roll speeds were the best. The best speed in this case was 30 rpm and a small roll opening of 0.010".

It was found that when fired UO_2 powder was used for the core material, the strips showed a tendency to split within the core. It was further found that when unfired -325 mesh UO_2 was used for core material, the splitting stopped.

Some work was started on closing the exposed ends of an experimental stainless-steel-clad foil element. Effective seals had been made last month on aluminum foil elements by rolling aluminum powder onto the ends of a green foil element strip and then completely densifying the powder by hot rolling the strip. This same method was attempted using stainless powder in aluminum foil. Because of the adherence of the aluminum foil to the stainless steel, however, the method was found to be unsuitable. An alternate method will have to be tried. It is proposed to use thin 0.001" steel shim stock and -325 mesh stainless steel powder for this purpose.

When this method is developed and the elements are sealed, X-ray and metallographic investigation will be used to check the UO_2 distribution in the core and ascertain the effectiveness of the sealing.

DECLASSIFIED

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

A. Savannah River Fuel Elements (W. G. Lidman 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 for evaluation by the Savannah River Laboratory was continued. A total of thirteen plates were processed.

Nine plates have been autoclaved at 175°C in saturated steam for 40 hours. Six of the nine passed the test and have been shipped to Savannah River for their tests. Several plates have been set aside for destructive testing in the future.

A complete report covering all test data on the plates shipped to E. I. duPont has been forwarded with the shipments.

B. MTR Fuel Elements (M. H. Binstock)

Objective: To develop manufacturing methods for the production of MTR fuel elements containing large amounts of fissionable material dispersed in a bonded matrix.

The development of methods to produce the brazed, box-type fuel element assemblies for MTR was continued. An assembly was prepared from plates made by the powder metallurgy preform procedure described previously. The brazing was not complete and it now seems imperative that a furnace which can be controlled more accurately than our present equipment must be obtained. As the method for producing plates satisfactorily has been developed the future work will concentrate on brazing technique and will await the new furnace equipment.

C. 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.

This program is a study of hydrostatic pressing of metal powders for uniform density throughout the compact. A new piston design for the small hydrostatic pressing cylinder has been prepared which eliminates the stress concentrations which were present in the first model. Machining and heat treatment of this piston has been completed. The small hydrostatic pressing cylinder with this new piston was calibrated to pressures up to 80,000 psi - this is approximately double the pressure which can be achieved in the large, four-inch bore hydrostatic cylinder.

0371229.030

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

It was noted that the rubber 'O' ring seal which was suitable at the lower pressures would not maintain a satisfactory pressure seal at the higher pressures. Other materials, such as Neoprene and Teflon, were evaluated, and it was found that a harder rubber with a durometer reading of 90 was the most satisfactory.

Delivery of the pressure intensifier which was ordered for use with the four-inch bore cylinder has been delayed. It is anticipated that this equipment will be received and accepted during the month of June.

D. Non-Destructive Testing (N. Grossman and S. Priceman)

Objective: To develop non-destructive testing methods applicable to fuel elements.

To determine the density of the products by non-destructive means, a gamma ray absorption density measuring instrument is being made by the Ohmart Corp. of Cincinnati. The samples which were shipped in April were evaluated by Ohmart in a mock set-up. According to the preliminary results, an auxiliary compensating device will be necessary to take care of the dimensional variations.

Other activities on this project were discontinued on May 20th when the project was closed out.

E. Product Evaluation of Flat Plates (N. Grossman, S. Priceman and J. Bara)

Objective: To evaluate the process and product of the 14-foot flat plate fuel element development.

The construction of the autoclave for the corrosion testing of 14-foot flat plates was completed during the month. After several trial runs, three test runs were made. During the second and third runs, one fuel element failed. The disposal of the destructive-test plate and cleaning of the autoclave and the surroundings proved to be a time-consuming job. In order to facilitate the disposal problem a new container rack is being designed which will confine the oxidation and minimize the after-cleaning. These newly designed facilities will greatly reduce the time required for autoclave testing.

Because of the extensive destruction of the plates it was impossible to determine clearly the cause and origin of these failures. In order to determine and trace the development and progression of failure, short two-foot plates with synthetic defects are being tested in a smaller autoclave. The approach is to drill various small holes through the aluminum cladding and periodically remove the specimens from the autoclave to observe the effects of the high-temperature steam.

Tests to determine the tensile strength of the bond of several plates were carried out by brazing rods to the sheath and machining through the bond. There was a good correlation with ultrasonic results which indicated good bonding. The stress at failure varied from 1840 to 4220 psi. Over the areas which indicated only a fair bond by ultrasonics the values varied between 920 and 2860 psi.

DECLASSIFIED

E. Mechanism of Dimensional Instability of Uranium under Irradiation
(L. L. Seigle and R. Resnick)

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

In the study of the anisotropy of diffusion of uranium to test a theory of dimensional instability, efforts are being made to measure the self-diffusion coefficients of uranium. As noted in last month's report, large grained uranium foil will be substituted for single crystals in these experiments. Considerable time has been spent on the preparation

DECLASSIFIED

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

of the foil, both in rolling and critically straining and annealing. Grains of almost one quarter inch diameter were finally grown in 0.008" sheet by annealing at 640°C for one week after introducing approximately 1% strain into the metal by bending. The degree of perfection of the grains will be checked by x-ray metallography. A trial run is now being made of the entire procedure involved in the diffusion experiment as required by the AEC before U²³³ will be made available to us. When the isotope is finally obtained a surface decrease counting technique will be employed in determining the rate of diffusion.

Since our theory of dimensional instability is based on the hypothetical movement of imperfections, the study of porosity formation in metals is being continued in order to obtain some information about the behavior of lattice vacancies. Experimental evidence has been obtained that finely dispersed oxide particles act as nuclei for precipitating vacancies in alpha-brass. Most of these inclusions can be removed from the metal by directional solidification in a vertical furnace, or by zone purification. The evidence that the nuclei are oxide particles is as follows:

1. A zone-purified specimen, which exhibited little porosity on dezincification, was remelted in air, in a high-purity graphite crucible. After dezincing, the porosity formed was equivalent to that found in commercial material.
2. Several specimens were analyzed for oxygen content using the vacuum fusion method. The commercial stock showed 0.284 w/o O₂, Bridgmanized stock, 0.072 w/o O₂, and a specimen from the middle of a zone-purified copper ingot, 0.012 w/o O₂. The difference in oxygen content between the commercial material and the others is certainly significant.

F. 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.

An investigation is being made of the dependence of sintering rate of certain oxides upon oxygen pressure. Efforts within the past month have been concentrated on calibration of the dilatometer for measuring shrinkage of compacts of uniformly sized Cu₂O particles at one temperature and oxygen pressure. An unexpectedly large amount of scatter was found, shrinkages from run to run under the same conditions varying by as much as a factor of four or five.

To eliminate this variation the equipment has been redesigned and rebuilt. The thermal output of the furnace was increased to shorten the heating-up period at the beginning of the run as much as possible. At

DECLASSIFIED

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

the same time the telescope was mounted integrally with the dilatometer tube to reduce error due to thermal expansion of the mount.

In spite of the variation found from run to run, certain conclusions may be drawn from the experimental data at hand. Sintering of the fine Cu_2O particles began immediately and possibly at very low temperatures during heating of the specimens. In practically all cases, shrinkage due to sintering was greater than thermal expansion so that specimens did not show the preliminary expansion often found during the sintering of metallic powders. Log/log plots of shrinkage versus time curved continuously and no one of the theoretical equations derived for the variously assumed mechanisms of sintering could be applied to the entire course of sintering.

At the present time, apparatus is being constructed for more thorough drying of the atmosphere used in the dilatometer, since certain observations suggested that some of the scatter in previous data was due to reaction of the oxide with water. On completion of this work a new series of calibration runs will be started.

G. Fundamentals of Diffusional Bonding (L. 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 oil diffusion pump for the bonding apparatus has finally arrived and is being incorporated into the system. The loading mechanism is being calibrated as follows: the stress-strain curve in compression of a specimen of annealed tool steel is being determined on the Baldwin-Southwark testing machine; subsequently, the specimen will be compressed by the arbor press loading mechanism, and the resulting load will be calculated from the strain data obtained from the strain gages attached to the specimen.

The study of the growth is being made of diffusion layers in Al-Ni specimens. A number of specimens which had been bonded under pressures of 5 and 11 tsi for two minutes in the temperature range 525 to 575°C, are being 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. The results obtained to date are incomplete; however, they suggest that diffusion initially takes place at a faster rate than that predicted by classical solutions of the diffusion equation, and then slows down to a rate somewhat less than that predicted.

A number of interesting metallographic observations have been made of the characteristics of the layers formed. After hot pressing, two layers are readily discernible under the microscope. As shown in report SEP-132, the maximum strength of the Al-Ni bond is markedly increased if

DECLASSIFIED

~~CONFIDENTIAL~~

-22-

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

the nickel-rich layer is eliminated by increase in pressure during bonding; the increase in pressure favors the growth of the aluminum-rich layer at the expense of the nickel-rich layer. If the hot-pressed specimens are annealed at atmospheric pressure, the reverse proves to be the case. After 45 hours at 600°C, the thickness of the aluminum-rich layer becomes very small, and the bulk of the total layer consists of the nickel-rich phase. It is also observed that the aluminum-rich layer is brittle and is easily cracked during metallographic polishing, whereas the nickel-rich layer appears to be quite ductile. The difference in strength of the two phases and the brittleness of one phase could thus account for two of the results found by the previous investigators: (1) the increase in maximum strength with the elimination of the weaker nickel-rich phase and (2) the variability in the tensile strength data due to the brittleness of the stronger aluminum-rich phase.

An initial attempt has been made to identify the nickel-rich and the aluminum-rich phases by means of x-ray diffraction studies. For this purpose, a sample was employed which had fractured during a diffusion anneal along the interface between the aluminum-rich and the nickel-rich layers. The nickel-rich surface was irradiated and x-ray spectrometer traces were made. Subsequently, a layer was removed from the surface of the specimen by a hand polishing operation on 2-0 paper, and another x-ray spectrometer trace was obtained. This procedure is to be continued so that the identity of phases existing at successively greater distances from the interface can be identified.

The phase at the fracture surface on the nickel-rich side has again been tentatively identified as Ni_2Al_3 . This identification is based both on the visual comparison of intensities of the various lines and on the presence of the 4.90 and 3.50 "d" spacing, which appears in Ni_2Al_3 and not in NiAl nor in Ni_3Al nor in Ni . The Ni_2Al_3 phase persists below the fracture surface, and it is not yet clear whether any additional intermetallic phases lie between the Ni_2Al_3 and the Ni . In this connection, it should be pointed out that it will be difficult to differentiate between NiAl , Ni_3Al , and Ni in the presence of Ni_2Al_3 since the "d" spacings overlap. However, careful analysis should make it possible to distinguish between the phases. Also, samples of the intermetallic phases are being prepared by vacuum melting in a high frequency induction furnace; when the samples are ready, they will be used as standards to expedite the x-ray diffraction analyses.

/ew

Staff Coordination of work under Contract AT-30-1 GEN-366
H. H. Hausner

~~CONFIDENTIAL~~

CONFIDENTIAL