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METALLURGY DIVISION QUARTERLY PROGRESS REPORT FOR PERIOD ENDING JANUARY 31, 1953

April 6, 1953

Oak Ridge National Laboratory, Tennessee

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METALLURGY DIVISION

QUARTERLY PROGRESS REPORT

for Period Ending January 31, 1953

J. H. Frye, Jr., Director

EDITED BY

W. H. Bridges

DATE ISSUED

APR 6 1953

OAK RIDGE NATIONAL LABORATORY
Operated by
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ORNL-69	Period Ending May 31, 1948
ORNL-407	Period Ending July 31, 1949
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ORNL-583	Period Ending January 31, 1950
ORNL-754	Period Ending April 30, 1950
ORNL-827	Period Ending July 31, 1950
ORNL-910	Period Ending October 31, 1950
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ORNL-1161	Period Ending October 31, 1951
ORNL-1267	Period Ending January 31, 1952
ORNL-1302	Period Ending April 30, 1952
ORNL-1366	Period Ending July 31, 1952
ORNL-1437	Period Ending October 31, 1952

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METALLURGY DIVISION QUARTERLY PROGRESS REPORT

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PHYSICAL METALLURGY OF REACTOR MATERIALS

Investigation of the effect of vanadium additions (up to 8.5%) and silicon additions (up to 0.1%) on the properties of thorium has shown that neither of these is a potent strengthener. The effect of heat treatment on the properties of the vanadium alloys will be investigated.

Expansivity measurements on thorium have not shown any discontinuity in the temperature range 1325 to 1450°C, where a sharp increase in electrical resistivity had been noted previously. Further refined tests are planned.

Radiation damage tests on thorium specimens are now under way in the LITR. Specimens of unalloyed thorium and of 99% Th–1% U^{235} alloy are being prepared for MTR exposure.

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Test work indicates that a ceramic coating containing 10% boron, developed in the Ceramic Laboratory, can be successfully applied to a thickness of 15 mils on mild steel that is up to $^1/_{16}$ in. in thickness. A peculiar banding structure was observed in quartz specimens irradiated with a cobalt source. In the ceramic fuel element studies, annular rings have been formed from a cermet composition containing 70% Cr–30% Al_2O_3; about 8% UO_2 is included in sandwich form in the compact. Further accomplishments on ceramic fuels include the development of a SiC-Si body by using special techniques; preparation of USi_2 bodies; and the development of a design for an in-reactor high-temperature test rig. Work was carried out on the preparation of ThO_2 and UO_2 bodies intended for reactor service. An investigation of the vapor decomposition method for the application of coatings to ceramics was started. An investigation was begun on the development of insulating end caps for Hanford-type slugs. A study of the physical structure of the BeO blocks was made as an aid to the interpretation of the BeO-NaK compatibility tests. Petrographic studies included the examination of fluoride mixtures and other combinations r Petrographic investigation of the monohydrate of UO_3 is in progress.

REACTOR COMPONENT FABRICATION

Cladding experiments are in progress to establish the technical feasibility of a reactor design that features the use of thorium-uranium alloy fuel elements. Preliminary work indicates that the thorium-rich alloy is easily prepared and can be readily hot or cold worked by conventional methods; also, that the alloy can be metallurgically bonded to both aluminum and zirconium for construction of flat-plate-type elements. Attempts are being made to clad the alloy with stainless steel.

Alclad plates with 98.8 wt % thorium and highly enriched uranium cores have been fabricated and forwarded to the MTR for irradiation testing.

Initial design and fabrication studies to improve the operational performance of the MTR are complete. Results indicate that a gain in excess reactivity of 3 to 4% is possible by judicious modifications in design and techniques of manufacturing the fuel elements.

It is proposed to replace the lower stainless steel section in the shim control rod with an aluminum extrusion to reduce fabrication cost. The change will not adversely affect either operational control or the mechanical stability of the rod.

PHYSICAL METALLURGY OF REACTOR MATERIALS

E. J. Boyle

THORIUM ALLOY DEVELOPMENT

J. A. Milko

The alloy development program for improving the corrosion resistance and mechanical properties of thorium has continued. Analyses for the thorium-vanadium alloys, the mechanical properties of which were reported previously,[1] are now available and are listed in Table 1.

Concurrently with the preparation of thorium-vanadium alloys, alloys of silicon with Ames thorium were prepared by arc melting. Results of the effect of small additions of silicon on the mechanical properties of Ames thorium are listed in Table 2.

Thorium-Vanadium Alloys. The effects of vanadium on thorium are shown in the data of Table 1. Additions of about 0.6% to 1.2% of vanadium to thorium seem to lower the hardness and strength properties. This appears to be either a deoxidation action on the thorium by the vanadium or a

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[1] J. A. Milko, *Met. Div. Quar. Prog. Rep. Oct. 31, 1952*, ORNL-1437, p. 4.

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removal of carbon from a probable solid solution by the formation of vanadium carbide.

Additions of vanadium above about 1.2% appear to harden the thorium. However, the rate of hardening, or, for that matter, of strengthening, is not too marked. Therefore, by disregarding any probable effects of heat treatment, it may be concluded that vanadium does not appear to be a potent alloying element for thorium.

Thorium-Silicon Alloys. Additions of small amounts of silicon to Ames thorium tend to increase the strength properties and hardness gradually. Ductility, as measured by per cent elongation and reduction of area, is lowered gradually by the addition of small amounts of silicon. The data are presented in Table 2.

It seems desirable at this time for additional alloys of higher silicon content to be prepared for study. Knowledge of properties of such alloys would be directly applicable to several problems relating to reactor material design.

PHYSICAL PROPERTIES OF THORIUM

R. E. Adams

Previous work indicated that a sharp increase in the electrical resistance of thorium occurs in the

TABLE 1. EFFECT OF VANADIUM ON THE PROPERTIES OF AMES THORIUM

Annealed for ½ hr at 750°C after cold swaging about 50%

CHEMICAL COMPOSITION (%)			TENSILE STRENGTH (psi)	YIELD STRENGTH AT 0.2% OFFSET (psi)	PROPORTIONAL LIMIT (psi)	ELONGATION IN 1.5 in. (%)	REDUCTION OF AREA (%)	ROCKWELL H HARDNESS
V	C	Th(a)						
0.39	0.038	Balance	30,900(b)	20,000(b)	16,200(b)	47.0(b)	57.0(b)	78
0.59	0.036	Balance	29,300(c)	17,900(c)	13,700(c)	57.0(c)	64.0(c)	65
1.17	0.033	Balance	27,400(c)	16,600(c)	13,700(c)	51.0(c)	62.0(d)	75
3.33(e)	0.040(e)	Balance	33,600(d)	16,300(d)	11,400(d)	34.0(d)	55.0(d)	81
6.30(e)	0.087(e)	Balance	39,500(b)	20,400(b)	12,100(b)	26.0(b)	40.0(b)	93
8.54	0.042	Balance	36,700(b)	20,200(b)	12,700(b)	28.0(b)	50.0(b)	100

(a)Also contains trace impurities of Be, Al, Fe, N, Ca, Mg, B, Cd, and Zn.
(b)Average of four tests.
(c)Average of three tests.
(d)Average of two tests.
(e)Values appear to be high and will be checked.

TABLE 2. EFFECT OF SILICON ON THE PROPERTIES OF AMES THORIUM

Annealed for ½ hr at 750°C after cold reduction of about 85%

CHEMICAL COMPOSITION (%)			TENSILE STRENGTH (psi)	YIELD STRENGTH AT 0.2% OFFSET (psi)	PROPORTIONAL LIMIT (psi)	ELONGATION IN 2 in. (%)	REDUCTION OF AREA (%)	HARDNESS	
Si	C	Th(a)						Rockwell H	VHN
0.032	0.032	Balance	25,700(b)	15,000(b)	9,600(b)	51.0(b)	54.0(b)	89(c)	65(c)
0.077	0.037	Balance	27,700(b)	16,700(b)	11,000(b)	44.0(b)	54.0(b)	94(c)	72(c)
0.110	0.041	Balance	29,800(b)	18,700(b)	12,700(b)	42.0(b)	48.0(b)	95(c)	72(c)

(a)Also contains trace impurities of Be, Al, Fe, N, Ca, Mg, B, Cd, and Zn.
(b)Average of two tests.
(c)Average of ten readings.

temperature range of about 1325 to 1450°C. In an effort to ascribe a reason for this, investigations are being made to learn whether any other properties of thorium undergo changes in this temperature region.

Preliminary experiments have been made to measure the coefficient of thermal expansion of thorium at high temperatures. The data obtained thus far indicate that no significant discontinuities occur in the thermal expansion vs. temperature curve between about 900 and 1600°C. Since the test methods were somewhat crude, the coefficients of expansion of thorium are not accurately known. Some improvements in technique have been adopted, and later tests with iron have indicated that the phase transformation between alpha and gamma iron, and between gamma and delta iron, are clearly indicated. Additional tests with thorium will be made in the near future.

RADIATION DAMAGE OF THORIUM

R. E. Adams R. H. Kernohan[2]

A study of the effects of neutron irradiation on the properties of thorium is being carried out. Although no information has been obtained from these experiments, as yet, the status and scope of the program will be described here.

LITR Irradiation Experiments. Several thorium test specimens are now being irradiated in the LITR at an approximate flux of 4×10^{12} neutrons/cm^2·sec. A detailed description of the specimens was given in ORNL-1366.[3] Irradiation of the cylindrical and the hardness test specimens started on August 19, 1952, and the tensile and impact specimens were put in the reactor on December 1, 1952. The specimens will be exposed for six months.

MTR Irradiation Experiments. Plans have also been made for irradiation of thorium in the MTR. Specimens will be irradiated at a flux of about 10^{14} neutrons/cm^2·sec. One set of specimens will be exposed for six months; a duplicate set will be exposed for one year. The specimens will be canned in capsules containing liquid NaK to provide good heat transfer between the specimens and the capsule walls.

Each set of specimens will contain the following samples:

1. two cylindrical test specimens, 0.204 in. in diameter and 4 in. long, which will be used to study changes in dimensions, dynamic elastic modulus, electrical conductivity, and thermal conductivity;
2. four impact test specimens, 0.204 in. in diameter and $4\frac{3}{4}$ in. long, each of which can be broken in four places;
3. twelve hardness test specimens, 0.204 in. in diameter and $\frac{1}{8}$ in. thick, which are for study of changes in hardness and possible effects of postirradiation annealing.

A test program is also being planned for investigating the effects of irradiation damage in thorium containing U^{235} as an alloying addition. Tentatively, it is planned to irradiate a 99% Th–1% U^{235} alloy, and to approximately duplicate the present experiments scheduled for MTR irradiation.

PROPERTIES OF PURE METALS

W. J. Fretague

The Mechanical Testing group has performed elevated-temperature tensile tests on specimens made from six induction-melted nickel rods, which were described in a previous report.[4] Variations in the impurity content of the individual melts affected the physical properties of the material to such a degree as to make the data noninterpretable for the purpose for which the experiments were designed. A purchase order has been placed with the Vacuum Metals Corp. for 20 ft of $\frac{7}{8}$-in.-dia "Nivac" (high-purity, gas-free nickel). This material is produced in 200-lb ingots, and therefore it is possible to obtain specimens that are more homogeneous than those obtained from individual melts. When this material is delivered the experiments will be repeated.

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[2] Solid State Division.

[3] R. E. Adams, *Met. Div. Quar. Prog. Rep. July 31, 1952*, ORNL-1366, p. 10.

[4] W. J. Fretague, *Met. Div. Quar. Prog. Rep. July 31, 1952*, ORNL-1366, p. 10.

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HRP METALLURGY

W. J. Fretague

E. C. Miller W. J. Leonard

SERVICE TO CORROSION AND ENGINEERING GROUPS

Examination of a sample holder of type 304 stainless steel has established the susceptibility of a sensitized, austenitic, stainless steel to intergranular corrosion attack in uranyl sulfate containing 100 g of uranium per liter at 100°C under crevice corrosion conditions that involved oxygen depletion of the solution.

A variety of samples of stainless steels have been prepared for the Static and Dynamic Corrosion Test groups in an effort to establish the effect of such metallurgical variables as mechanical working, heat treatment, and various descaling procedures.

Valve parts of titanium and special stainless steels have been subjected to hard-surfacing treatments, exposed to corrosion conditions in dynamic corrosion loops, and evaluated metallurgically. This work is continuing because completely satisfactory treatments have not been established.

WELDING OF TYPE 347 STAINLESS STEEL

Experimental work has been carried on in the welding of thick plates of type 347 stainless steel by using various combinations of passes, by using bare rod filler metal in the inert-gas shielded-arc method, and by using coated electrodes in the conventional, metal-arc welding method. Similar work has been done in the welding of type 347 stainless steel to a boiler grade of carbon steel. Tentative procedural specifications and a welder's qualification outline have been written for the heavy-section welding of type 347 stainless steel.

RADIATION DAMAGE STUDIES

Assistance has been given to the Solid State Division in the selection and preparation of impact specimens for exposure in the Oak Ridge graphite reactor and in the LITR. The materials prepared included several carbon steels, austenitic stainless steels, and titanium of various grades.

PROPERTIES OF TITANIUM

Vacuum-fusion analyses and impact tests have been performed on specimens of commercial-purity titanium, as well as on high-purity iodide titanium – as machined, as exposed to uranyl sulfate under static and under dynamic corrosion conditions, and after cathodical treatment – for the purpose of investigating the possibility of contamination and embrittlement by hydrogen during the course of reactor operation.

The limited results available to date are encouraging in that they do not indicate that uranyl sulfate corrosion adds any appreciable amount of hydrogen to commercial-purity titanium or that it becomes any more embrittled as a result of the corrosion. It should be remembered, however, that the tests are not sufficiently complete to be conclusive and that the commercial-purity titanium was rather brittle even before the efforts to introduce hydrogen.

FABRICATION

E. S. Bomar

HRE Recombiner. Operation of the HRE results in a continuous dissociation of the heavy-water moderator into deuterium and oxygen. One method of recombining these gases for return to the reactor makes use of a controlled catalytic reaction to reform heavy water. J. Ransohoff of the Reactor Experimental Engineering Division suggested the possibility of applying the catalyst to a metallic surface, which could serve as the high-temperature side of a heat exchanger. The need for a large reacting area led to the work described below.

The structure of metal built up by using a wire or powder spray gun is inherently porous and has a relatively rough finish. Since these properties provide increased surface area, it was decided to investigate the possibilities of these methods of fabrication. Experimental lengths of ¼-in.-OD tubing were prepared by spraying stainless steel on stainless steel tubing to a thickness of 0.010 inch. Bends of 180 deg caused separation of the sprayed layer from the tube. To minimize this, a thin layer of a brazing alloy was applied to the tube before spraying.

Approximately 0.005 in. of Nicrobraz alloy was applied to the tube by using a powder pistol. This

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was covered with a 0.020-in. layer of type 302 stainless steel from a wire spray gun. The composite tube was then sintered at 1120°C in hydrogen. Since this sintering temperature is above the melting point of the Nicrobraz, a bond was created between the tube and the sprayed metal layer. The adherence during bending was much better than without the braze. Thirty tubes, ½ in. in outside diameter and approximately 10 to 12 in. long have been prepared for assembly into a small heat exchanger by the Reactor Experimental Engineering Division.

Riveting of Second HRE Impeller. A series of five rivets for a second titanium impeller were upset by using conditions established in assembling the first impeller.[1] The riveting proceeded without difficulty, and all rivets upset in a satisfactory manner, as indicated by a subsequent cleanup machining operation. All of the rivets were of the same length. The conditions for riveting are given in the following:

Rivet length, in.	1³⁄₆₄
Rivet distance, in.	0.178
Voltage on electrodes	4.20
Current, %	75
Approximate current, amp	1000
Time in cycles	50

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[1] E. S. Bomar, *Met. Div. Quar. Prog. Rep. July 31, 1952*, ORNL-1366, p. 18.

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CERAMICS RESEARCH

J. M. Warde

C. E. Curtis	J. R. Johnson
L. M. Doney	A. J. Taylor
S. D. Fulkerson	G. D. White
J. A. Griffin	

T. N. McVay, Consultant
[redacted]nsultant

[redacted]

RADIATION DAMAGE STUDIES

Further work in the radiation damage studies awaits the results of exposures of ceramic specimens at Hanford. Some additional studies of the banding observed in specimens of quartz irradiated with a cobalt source were made (Fig. 17). It is believed that these bands are *f*-center phenomena resulting from trace impurities in the quartz. It is possible that some correlation of geologic history with the size and nature of the bands may be found.

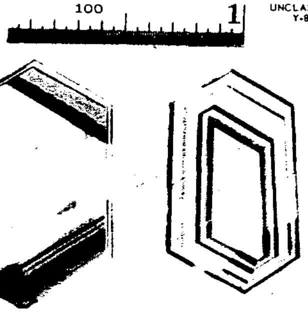

Fig. 17. Quartz Specimens After Irradiation with a Cobalt Source. Specimen on right from Brazil; specimen on left from Arkansas.

DEVELOPMENT OF CERMETS FOR REACTOR COMPONENTS

The Cr-Al_2O_3 cermet fabrication studies have esulted in the successful preparation of disks in. in diameter and 20 mils thick. Several sand- ich disks were also made that contained a thin yer of UO_2 mixed with 70% Cr–30% Al_2O_3. nnular rings have been formed of the 70% Cr–30% $_2O_3$ composition containing about 8% UO_2 in ndwich form. In-reactor tests of these cermets e now being contemplated. A ZrC-Fe cermet was fabricated as a possible material for pump parts, seals, and bearing materials. This cermet, along with Kennametal 151-a, has been tested for corrosion resistance in fluorides, sodium, and lead. These materials appear to be very promising. The Kennametal 151-a showed no attack at 800°C for 100 hr in sodium, lead, and fluoride No. 14. A slight surface leaching was observed on the ZrC-Fe cermet in both sodium and fluoride No. 14.

The development of fuel elements using combinations of SiC-Si and USi_x was started. The SiC-Si body is prepared by heating at 1000°C for 1 hr a mixture of 50% graphite (2 micron) and 50% wheat flour. The resulting porous body is then placed in a graphite container together with powdered silicon, and the container is heated by resistance to 2000°C, at which temperature silicon carbide is formed, with an accompanying exothermic reaction, while molten silicon is simultaneously absorbed by the body. The resulting cermet appears to have a well-bonded structure that is free from voids. Further work is in progress to determine the physical properties of this material.

An investigation of the preparation of USi_x has commenced. A material containing about 90% USi_2, 5% USi_3, and 5% UO_2 has been successfully prepared by heating uranium metal in contact with silicon at 1700°C. It is necessary to have a very pure argon or helium atmosphere for this reaction.

Preliminary designs of an in-reactor high-temperature test rig were made. It is proposed that a design be prepared for a small chamber about 4 in. in outside diameter and 12 in. long with two concentric compartments; the inner compartment will be for containing ceramic fuels, and the outer compartment will contain water cooling coils that will serve to cool both the container and any hot gases from the inner fuel element chamber. The following gas cycle is to be used: out-of-reactor pump to inner chamber to outer chamber to out-of-reactor radioactive counting rig to out-of-reactor pump. The out-of-reactor pump is to be used to provide flow rates over the fuel element of the order of 200 fps.

OXIDE CERAMICS

Thorium Oxide. In determining the properties of thorium oxide for possible application as a reactor material, it was shown that firing temperatures of 1400 and 1600°C (held for 1 hr) are insufficient to vitrify pure thorium oxide. Firing at 1800°C and the effects of additions of alumina, silica, zirconia, and of other ingredients in promoting vitrification at lower temperatures are being investigated. Measurements of the resistance of thoria to autoclaving are being conducted through R. B. Briggs' HRE Group, with the use of fused thoria pellets obtained by this laboratory from the Norton Company.

Three thoria cylinders (solid) 2 in. high and 1 in. in diameter were slip-cast and fired at 1600°C for the *Experimental Engineering Department*. The cylinders are to be used in testing the thermal conductivity of thorium oxide at room temperature.

Uranium Oxide. Hot-pressed specimens of uranium oxide have been transmitted to R. B. Briggs, HRE Group, for autoclave testing.

FABRICATION OF SPHERES FROM CERAMIC MATERIALS

A laboratory method for producing small (1/8 to 1/2 in. in diameter) spheres from powdered ceramic materials is being worked out. This technique will be employed for the production of uranium oxide pellets and will be useful for various other applications.

VAPOR COATING OF BERYLLIUM OXIDE

The coating of graphite by the vapor decomposition method has been described by Kertesz *et al.*(1) Because hot-pressed beryllium oxide samples have, in the past, shown a tendency toward particle disintegration when dynamically tested in liquid NaK, it was thought that a coating of silicon, silicon carbide, or some of the refractory metals might retard, if not completely stop, this disintegration. Accordingly, an apparatus for vapor deposition experiments has been rigged and an investigation commenced under the joint auspices of the Ceramic Laboratory and the Chemistry Section. So far, it has been established that hot-pressed beryllium oxide specimens can be coated by this method by passing $SiCl_4$ vapor in a hydrogen atmosphere over heated specimens of beryllium oxide. It was found that unless the specimen temperature is kept in the range 900 to 950°C, and the flow of vapors is kept at a very low rate, the coating is deposited too rapidly and it is too thick and too uneven. Such a coating spalls off readily when cooling. For silicon carbide coatings, in the preparation of which $SiCl_4$ and hydrogen vapors are first bubbled through liquid toluene and then passed over the heated sample, the same operating conditions hold. This popping off of the heavy coating is due, in part, to the wide difference in the coefficients of expansion of the materials involved. A specimen of silicon that was successfully coated with a thin and uniform coating was submitted

or a 200-hr dynamic test in liquid NaK. Results of this test are not yet available. Also, a specimen of beryllia presumably

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(1)F. Kertesz, H. C. Brassfield, and L. M. Doney, *Coating Graphite with Silicon and with Silicon Carbide by the Vapor Decomposition Method*, NEPA-1553 (Aug. 1, 1950).

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coated with silicon carbide has been submitted for x-ray determination. It is planned to try coatings of zirconium, molybdenum, titanium, deposited in a similar manner, by using a volatile compound of these metals as a source.

INSULATING END-CAP DEVELOPMENT

Work has commenced on developing insulated end caps for Hanford-type slugs. It is proposed to fasten disks of ceramic materials inside of the aluminum end caps during the slug canning operation. The purpose of the caps will be to equalize the heat flow from the center of the slug to the surface. A number of disks of various ceramic materials, such as SiO_2 glass, forsterite, zircon, and alumina porcelain, have been prepared. These disks are 30 mils thick and 1.1 in. in diameter. It was originally intended to make up canned slugs with the ceramic disks in place and forward them to Hanford for tests. Unfortunately, the Y-12 canning line has been shut down and the expense of setting up to get the job done here is prohibitive. The disks have therefore been sent to Hanford for make-up of the slugs.

UNIFORMITY OF BERYLLIUM OXIDE BLOCKS

A study of the physical structure of the beryllium oxide blocks to be used [redacted] was made to aid in the interpretation of BeO-NaK compatibility test data. These blocks, fabricated by the Norton Company, were made by hot-pressing beryllium oxide powder. They are hexagonal blocks $2\frac{1}{8}$ in. on a side, $3\frac{11}{16}$ in. in diameter across the center of the faces, and 6 in. long. They are fabricated with a center hole running axially through the block; the diameter of the hole (depending on the position of the block in the reactor core) is $\frac{1}{2}$, $1\frac{1}{8}$, or $1\frac{3}{4}$ inches. A visual examination was made of about 30 blocks of the type having a $\frac{1}{2}$-in.-dia hole. These blocks had been sawed in two on a plane perpendicular to the long axis. It was observed that in all cases the core of the blocks had a different physical structure than that of the outer portion of the same blocks. In order to study the core structure, six blocks that had not been sawed were soaked in fuchsin dye for 12 hours. The dye is absorbed most in the more porous areas in the block; Fig. 18 shows the typical soft-core structure noted in all the dyed specimens. Samples taken from the inside and outside surfaces of these blocks showed density variations from 2.80 to 2.83 at the outside, which decreased to 2.26 to 2.43 at the inside. The apparent porosity of the dense portion of the block is practically zero, compared with values up to 23.3% for the soft core. Table 25 gives results of density, apparent porosity, and water absorption measurements for specimens cut from various portions of the block.

Blocks that have a $1\frac{1}{8}$-in.-dia central hole are being split longitudinally so that the two halves can be placed around a cooling pipe. With the blocks split, a large amount of inner surface will

TABLE 25. DENSITY, POROSITY, AND WATER ABSORPTION MEASUREMENTS OF VARIOUS SECTIONS OF THE BERYLLIUM OXIDE BLOCK

SOURCE	WET WEIGHT, W (g)	DRY WEIGHT, D (g)	$W-D$	ABSORPTION, $\frac{W-D}{D}$ (%)	SUSPENDED WEIGHT, S_Δ* (g)	$W-S_{\Delta\Delta}$**	DENSITY (g/cm^3)	POROSITY $\frac{W-D}{W-S}$ (%)
Outer edge	1.3612	1.3609		0	0.881	0.4802	2.83	0
	1.8459	1.8458		0	1.187	0.6589	2.80	0
	2.0859	2.0860		0	1.350	0.7359	2.83	0
Core	1.3957	1.2789	0.1168	9.13	0.846	0.5497	2.33	21.25
	1.3555	1.2661	0.0894	7.06	0.835	0.5205	2.43	17.18
	1.1778	1.0680	0.1098	10.28	0.707	0.4708	2.27	23.32

*S_Δ = weight suspended in water.

**$S_{\Delta\Delta}$ = equivalent to volume.

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Fig. 18. Dye-treated Beryllium Oxide Bloc Showing Porous Central Section.

be exposed to the coolant material. This inne surface is low-density material that is particularl susceptible to NaK corrosion. To determine th extent of the low-density region, a random sampl of these blocks was soaked in fuchsin dye fo 12 hr and specimens were cut from selected localities in the block. Figure 19 shows the densit and per cent porosity placed on the block in th location from which the sample was taken. Th dark shaded areas on the right of the block indicat the porous portions shown up by the penetratio of the dye. Five samples of blocks with a $1\frac{3}{4}$-in.-dia central hole were soaked in dye and were als found to have a porous central core.

A general observation was made regarding th prevalence of cracks in all the blocks examined. All the blocks have at least one crack, and some blocks contain several cracks. In some cases these cracks are visible at the surface of the block, but in the majority of the blocks the cracks are confined to the interior portion. These cracks are quite narrow and contribute only slightly to the porosity. The presence of one of these cracks may be observed in Fig. 19.

The porous condition developed in the hot-pressed beryllium oxide blocks is a defect caused by fabrication. A probable cause of the defect is that the hot-pressed die had too tight a fit on the top and bottom punches around the central pin, which would cause binding and therefore uneven pressure application. A further factor is faulty filling of the die, which would cause bridging of the powder around the pin. Other factors, such as too short time of application of pressure, too short time of holding at pressing temperature, uneven temperature distribution in the beryllium oxide powder because of too rapid heating, cooling of the die to room temperature without extracting the core pin, etc., could also have affected the final density.

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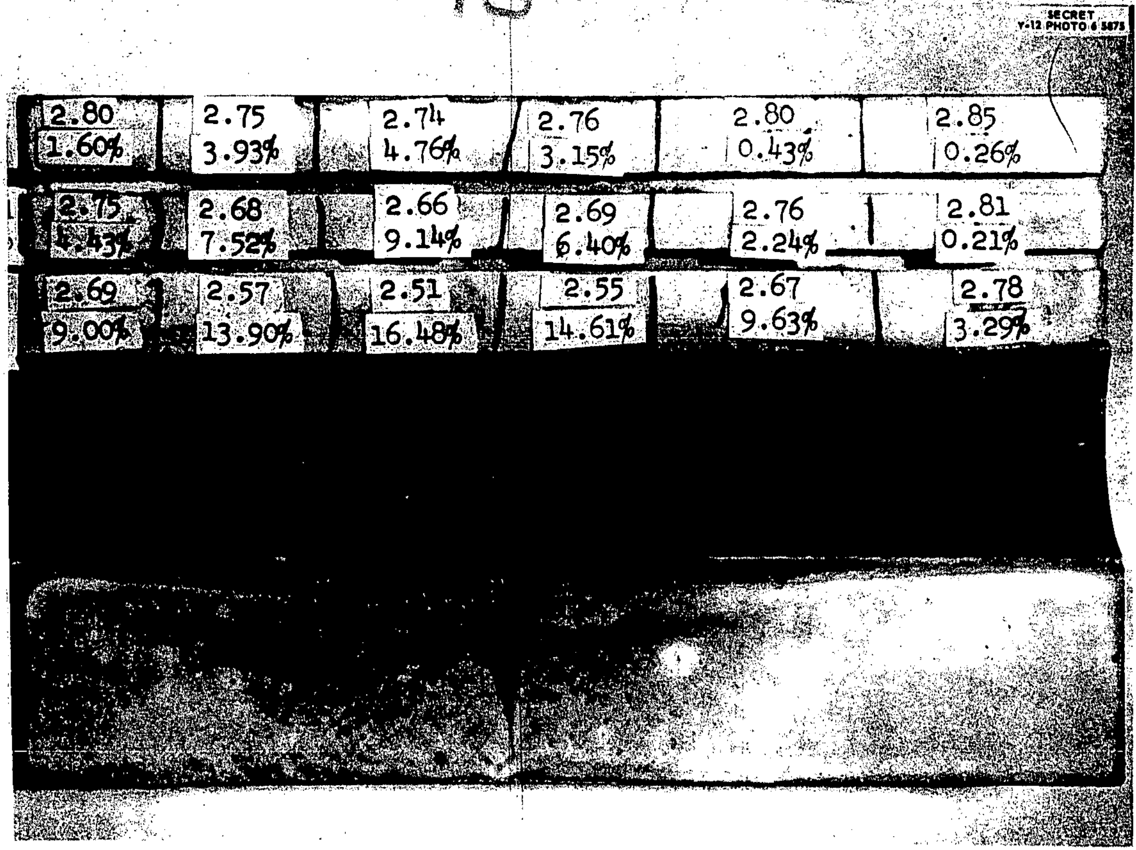

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Fig. 19. Density and Per Cent Porosity at Various Locations in a Full-Size, Beryllium Oxide Block.

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REACTOR COMPONENT FABRICATION

J. E. Cunningham

THORIUM-URANIUM FUEL ELEMENT

R. J. Beaver J. W. Geer

Fabrication studies were initiated on a converter-breeder type of thorium-uranium alloy fuel plate for possible service in a H_2O-cooled and -moderated "package" reactor. There is interest in such a fuel element because the long service life obtainable would result in substantial savings in both fabrication and chemical processing costs, as well as in fuel investment.

Although when in use there will be an ultimate need for either zirconium or stainless steel cladding, the initial work was done with aluminum cladding, primarily to expedite radiation damage testing of the alloy in the MTR. The main reasons for the choice of aluminum are (1) it avoids the use of dissimilar metals in intimate contact in the same system or the possibility of galvanic corrosion, and (2) it can readily be stripped chemically from the alloy to facilitate dimensional and physical property measurements after irradiation.

Several melts of thorium-rich alloys, 1 to 5 wt % uranium range, were successfully prepared in the arc-melting furnace. Chemical analysis verified the intended composition, within the accuracy of the determination.

The alloy can be readily hot or cold worked. Room temperature hardness values of the alloy are given in Table 26.

The alloy can be metallurgically bonded to commercial 2S-grade aluminum by hot rolling in the 400 to 450°C temperature range with a total reduction in thickness of 8 to 1. Jacketed billets rolled at temperatures in excess of 450°C show the presence of the brittle $ThAl_3$ compound at the alloy-aluminum interface. It is not necessary to evacuate jacketed billets that are rolled below 450°C.

Like unalloyed thorium, the uranium-thorium alloy tends to "ball-up" on the leading and trailing ends when rolled in a composite pack with aluminum cladding. The resulting nonuniformity in

TABLE 26. ROOM-TEMPERATURE HARDNESS OF THORIUM-RICH ALLOYS

ALLOY COMPOSITION	CONDITION	HARDNESS IN DPH (10-kg load)
Unalloyed Ames thorium metal	Cold worked 30%	99
1.1% U–Th	Cold worked 33%	116
1.1% U–Th	Cold worked 33% and annealed 6 hr at 800°C	89
1.1% U–2% Zr–Th	Cold worked 33% and annealed 6 hr at 800°C	69

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core thickness is believed to be due to the difference in plasticity between the two materials at rolling temperatures.

This defect can be partly corrected by using either 24ST- or 52S-grade aluminum to back up or stiffen the 2S-grade aluminum cladding. However, this results in either a 5- or 7-ply sandwich, depending on whether or not 2S-grade aluminum is desired as the outer cladding material.

Three Alclad, enriched, uranium-thorium alloy plates, 8 in. long, 1 in. wide, and 0.072 in. thick, and one plate 28 in. long, 1 in. wide, and 0.072 in. thick, were fabricated for irradiation testing in the MTR. The alloy core used in the preparation of these plates was composed of 98.8% thorium and 1.2% highly enriched uranium metal.

The cladding techniques developed and proved to be successful for cladding Ames thorium metal with zirconium were tried on a 4% uranium-thorium alloy. The brazing and the diffusion-anneal cladding techniques appear to work equally well on this thorium-rich alloy.

The brazing-in-vacuum technique, in which a brazing alloy of 22% zirconium-thorium alloy is used, was fully described in a previous report.(1) The diffusion-anneal method consists of picture framing the alloy in zirconium and canning the entire compact in a stainless steel jacket for oxidation protection during hot rolling. After evacuation through an exhaust tube, the assembly is sealed by hot forging. The use of NBS ceramic frit A-418 coated on the stainless steel prevents the formation of the iron-zirconium eutectic during the diffusion-anneal treatment.

FABRICATION OF URANIUM-ALUMINUM ALLOY COMPONENTS

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MTR Experimental Studies. Work on the problem of improving the operational performance of the MTR continues. Techniques have been found that minimize the amount of poisonous brazing flux entrapped in the fuel element during manufacture. The V_{Al}-to-V_{H_2O} ratio can be safely lowered by cautious modifications of the fuel element design. The proposed substitution of an aluminum extrusion for the lower stainless steel bearing section in the shim control rod will not adversely affect either operational control or the mechanical stability of the rod.

Results of this work were discussed in detail with members of the Phillips Petroleum Co. and the Idaho Operations Office at a meeting held in December.(2) Recommendations were (1) that one control rod of the new design be constructed and tested at ORNL, (2) that an experimental loading of 23, thin, side-plate units be fabricated according to the latest techniques to be ready for the next reloading on about February 1, 1953, and (3) that the Phillips Petroleum Co. investigate a fuel assembly design that features more fuel concentrated in the upper half of the element than in the lower half.

Studies to Eliminate Flux Entrapment. Although not known with certainty, it was strongly suspected that the fuel element was being poisoned by simple mechanical entrapment of brazing flux, and experimental work on the problem of removal followed this lead. Another possible mechanism was that of lithium alloying with the aluminum-silicon braze metal in much the same manner that sodium is used to modify this particular alloy.

Experimental work was conducted in two steps. The initial phase consisted of investigating the effect of higher brazing temperature, joint design, and techniques of applying the flux. No attempt was made to alter the composition of the flux. This work is now complete and the important results are summarized in Table 27.

All tests were run on full-sized, dummy, aluminum fuel units. After brazing, the units were carefully dissolved by the Chemical Technology Division and the solution was analyzed for lithium content by the flame photometer method. The accuracy of the determination is better than 2 ppm at a 95% confidence level. In all but the first test, brazing flux from the same batch was used to remove the variable of change in flux composition. Analysis showed that this flux contained 4.72% lithium and 54.0% chlorine.

The amount of chlorine and flux contained in each unit (shown in Table 27) was calculated on the basis of the above analysis. The use of mercuric nitrate as a catalyst in the dissolving

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(1) J. E. Cunningham *et al.*, *Met. Div. Quar. Prog. Rep. July 31, 1952*, ORNL-1366, p. 14.

(2) J. E. Cunningham, *MTR Fuel and Control Rod Element Meeting*, ORNL CF-53-1-150 (Jan. 14, 1953).

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TABLE 27. RESULTS OF BRAZING TESTS TO ELIMINATE FLUX ENTRAPMENT

TEST ASSEMBLY	BRAZING CYCLE		AMOUNT PER UNIT (g)			CALCULATED LOSS IN $\Delta k/k$ IN ACTIVE LATTICE DUE TO		
	Temperature (°F)	Time (min)	Li	Cl	Flux	Li	Cl	Li + Cl
X-1	1100	37	1.26	14.41	26.69	2.52	2.67	5.19
X-13	1105	35	0.57	6.52	12.08	1.13	1.22	2.35
X-21*	1105	33	0.17	1.94	3.60	0.35	0.37	0.72
X-22*	1115	31	0.09	1.04	1.93	0.19	0.20	0.39
X-23**	1115	31	0.06	0.76	1.40	0.13	0.14	0.27

*Side plate, $\frac{1}{8}$ in. thick, with shallow groove joint.

**Same as X-21 except for change in technique of applying flux.

step prevented a direct check on the chlorine content in the dissolved assembly solution.

The values of loss in $\Delta k/k$ were calculated on the basis of a 3- by 9-slab loading arrangement of 140-g elements with a total poison cross section of 7700 cm^2.

The results of the brazing tests show that the combination of higher brazing temperature, more shallow groove joint, and flooding of the side plate with an excess of flux reduced the amount of lithium, and presumably chlorine, by a factor of 21. Attempts to braze at 1120°F were not successful because the braze metal alloyed excessively with the 2S-grade aluminum side plate at this temperature.

In the second phase of the investigation, the work was centered on the problem of developing a new brazing flux that is essentially free of lithium and chlorine. Several special fluxes prepared by Alcoa and Permalatem were tested, with unsuccessful results.

A new bromide-base flux, developed at ORNL, showed promise during initial testing. The flux is prepared from a base mixture of 20 parts of barium bromide, 60 of calcium bromide, and 20 of sodium bromide, with 2 parts of either $CaCl_2$ of $SrCl_2$ and $1\frac{1}{2}$ parts of $ZnCl_2$ added. Testing is incomplete.

Design and Fabrication Studies to Improve the Al-to-H_2O Ratio. Another approach to enhance the performance of the reactor is to alter the fuel element design to obtain a lower V_{Al}-to-V_{H_2O} ratio in the active core. Structurally, the present fuel element is over-designed for service in the MTR. Study revealed, however, that any contemplated design change is limited largely by the hydraulics of the present system; hence, only minor changes in the fuel element were considered. The modifications considered were guided, generally, by the following factors: (1) an Al-to-H_2O ratio in the active core of less than 0.60 would be desirable; (2) the external dimensions of the fuel unit would remain the same; (3) any fuel component member altered would not be substantially weakened structurally; (4) effects on water velocity and flow area would be investigated; (5) no similar change would be made in the fuel section of the shim control rod; (6) manufacturing cost would not increase.

The Al-to-H_2O ratio of a single fuel element of the existing design ($\frac{3}{16}$-in.-thick side plate and 0.060-in.-thick fuel plates) is 0.716. This ratio increases slightly to 0.731 for the entire 3- by 9-slab loading arrangement because of the additional aluminum in the four shim control rods.

The first change investigated was that of reducing the side-plate thickness from $\frac{3}{16}$ to $\frac{1}{8}$ inch. This lowered the Al-to-H_2O ratio in the active lattice to 0.689 and increased k_{eff} by approximately 1%. The water flow area increased 2.5%

A dozen or more full-sized units of this design were successfully mocked up according to specifications, and no manufacturing difficulties were found. Subsequently, an enriched fuel element of the same design was fabricated and transferred for service in the LITR.

The second modification considered was that of decreasing the cladding on the fuel plate from 20

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to 15 mils. The combination of this change plus the thin-side plate drops the Al-to-H_2O ratio in the active lattice to 0.569, but the water gap increases from 117 to 128 mils.

No attempt was made to fabricate this particular unit because of the objectionable increase in water-gap thickness. Instead, another fuel plate was added to the unit to bring the water-gap thickness back to 118 mils.

A 3- by 9-slab loading of these 19-plate elements has an Al-to-H_2O ratio of 0.600, and, more importantly, it increases k_{eff} by approximately 4%. A portion of this gain, however, is due to the extra fuel present because of the added plate. The water-flow area is increased appreciably, that is, by 8.2%.

It should be pointed out that this change does not materially weaken the fuel plate structurally, because the strength of the member is determined largely by the core alloy, which has been altered. The yield strength of annealed 2S-grade aluminum, for instance, is 5000 psi, whereas the 17% uranium-aluminum alloy has a yield strength of 9000 psi, based on a 0.2% offset at room temperature.

The change does proportionately lower the amount of corrosion protection afforded. It is felt, however, that 15 mils of cladding would be more than adequate to protect the alloy core, which, by itself, exhibits fair corrosion resistance to water. Examination of elements at ORNL after ten months of service in the Bulk Shielding Reactor failed to reveal any pitting type of attack or other corrosion phenomena. Service in static water, such as in the BSR, is more severe corrosion-wise than is the 30-fps water velocity condition that exists in the MTR.

Neutron-wise, it is certainly advantageous to replace aluminum with water. Furthermore, only 1 to 2 mils of aluminum is required to stop secondary neutron recoils.

Construction of the 19-plate element presents no manufacturing problems. Several full-sized units were prepared to establish the dimensions, and each unit met specifications in all respects. Also, an enriched fuel element was fabricated for service in the LITR.

The possibility of lowering the aluminum cladding to 10 mils was checked, but analysis revealed that the water-flow area would increase appreciably, that is, by 14.5%.

Results of this and the other modifications discussed are summarized in Table 28. For comparative purposes, cross-sectional views of the present, thin side-plate units and of the 19-plate fuel element are shown in Fig. 20.

The cladding was dropped in increments of 5 mils so that the commercially available, aluminum-stock gages could be used in the Alcladding operation. Actually, the cladding could be reduced in increments of, say, 1 mil but this would require an additional rolling operation and, hence, a greater cost per unit.

Proposed Change in the Lower Stainless Steel Section of the Shim Control Rod. It is proposed to replace the lower stainless steel section in the shim control rod with an aluminum extrusion to reduce fabrication cost. This lower bearing section is presently being fabricated from type 304 stainless steel flat stock that must be machined, welded, stress-relieved, and remachined prior to assembly. Replacing this item with a 63S-grade aluminum extrusion would eliminate most of the costly labor operations.

The effect of this change on operational control, as well as on the mechanical stability of the new rod, was investigated by Gall.(3) Changes in magnet release and drop time were calculated. Also, the stresses produced in the proposed aluminum member by shock on the rod when dropped with full water flow were checked by two methods of analysis.

In conclusion, the study predicted that the change would: (1) decrease the fabrication cost by approximately $300; (2) increase the drop plus magnet release time, under conditions of no water flow, by 10%; (3) produce a stress in the aluminum extrusion, caused by shock on the rod when dropped at full water flow, of less magnitude than exists in the bottom of the fuel section of the existing design. These predicted effects should be confirmed by conducting drop tests. Specifications on the new, aluminum, lower section are given in engineering drawing TD-3038.

MTR Replacement Fuel and Control Rod Elements. The 13 reactor loadings of standard ($^3/_{16}$-in.-thick side plates and 60-mils-thick active plates) enriched fuel and control elements on order for calendar year 1952 were completed according to specifications and shipped to Arco.

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(3) W. R. Gall, *MTR Shim Rod Aluminum Lower Bearing Section*, ORNL CF-52-11-216 (Nov. 29, 1952).

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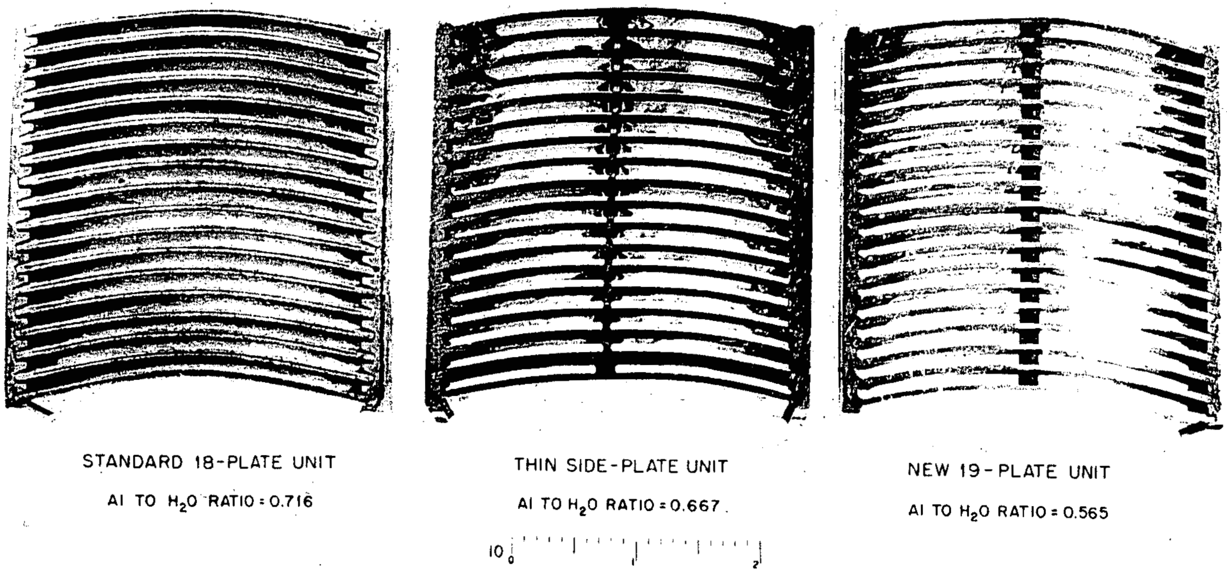

STANDARD 18-PLATE UNIT

Al TO H_2O RATIO = 0.716

THIN SIDE-PLATE UNIT

Al TO H_2O RATIO = 0.667

NEW 19-PLATE UNIT

Al TO H_2O RATIO = 0.565

Fig. 20. Cross-Sectional View of Present and Modified MTR Fuel Assemblies.

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Shipment on the last loading was made early in December. In addition, three, spare, beryllium-cadmium control rods were fabricated and forwarded.

An experimental loading of "sweetened" fuel elements was completed and shipped to the site in time for the scheduled February 1, 1953 reloading date. These units were fabricated according to specifications determined in discussions at the December meeting; ⅛-in.-thick aluminum side plates with shallow-groove joints were used, and the side plates were flooded with an excess of flux and brazed at a slightly higher temperature than that normally used.

Inspection revealed that all 23 units were well within dimensional tolerance. The maximum deviation of water-gap spacing, for instance, was approximately 117 ± 0.006 mils, or to within ±5% of the specified spacing and the average deviation was about ±3%.

Fuel and Control Rod Elements. The initial loading of enriched uranium-aluminum alloy

TABLE 28. AREAS AND VOLUMES OF MATERIALS IN THE MTR ACTIVE LATTICE FOR SEVERAL FUEL UNIT DESIGNS FOR THE 3- BY 9-SLAB LATTICE 23.622 in. (60 cm) IN LENGTH

Fuel Unit Description						
No. of fuel plates	18	18	18	19	19	20
Side-plate thickness, in.	0.188	0.125	0.125	0.125	0.125	0.125
Cladding-core-cladding thickness, mils	20-20-20	20-20-20	15-20-15	15-20-15	10-20-10	10-20-10
Water-gap spacing, in.	0.117	0.117	0.128	0.118	0.129	0.118
Al-to-H_2O ratio	0.716	0.667	0.529	0.565	0.439	0.464
Areas and Volumes of Materials in Lattice*						
Aluminum						
Area, in.^{2}	111.747	107.929	95.923	99.189	86.861	89.460
Volume, in.^{3}	2639.688	2549.499	2265.893	2343.043	2051.831	2113.224
Water						
Area, in.^{2}	152.797	156.615	168.621	165.332	177.660	175.015
Volume, in.^{3}	3609.371	3699.560	3983.165	3905.473	4196.685	4134.204
Increase in area, %		2.5	10.4	8.2	16.3	14.5
Al-to-H_2O ratio	0.731	0.689	0.569	0.600	0.489	0.511
U^{235}						
Area, in.^{2}	0.607	0.607	0.607	0.628	0.628	0.671
Volume, in.^{3}	14.339	14.339	14.339	14.835	14.835	15.850
U^{234}						
Area, in.^{2}	0.007	0.007	0.007	0.007	0.007	0.008
Volume, in.^{3}	0.165	0.165	0.165	0.165	0.165	0.189
U^{238}						
Area, in.^{2}	0.037	0.037	0.037	0.039	0.039	0.041
Volume, in.^{3}	8.740	8.740	8.740	9.213	9.213	9.685
Total uranium						
Area, in.^{2}	0.651	0.651	0.651	0.674	0.674	0.720
Multiplication Constants						
k_∞	1.682	1.079	1.6711	1.685	1.677	1.702
k_{eff}	1.218	1.227	1.253	1.256	1.277	1.289

*Total area in active core = 9.2137 × 28.7827 = 265.195 in.^{2}
Total volume in active core = 265.195 × 23.622 = 6264.44 in.^{3}

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fuel elements [redacted] s been manufactured and packaged for shipment. The loading consists of 30 of the standard, Bulk Shielding Reactor type of fuel element, 4 special fuel sections to house control rods, and 3 partial units containing 5, 9, and 14 active plates.

Unfortunately, delivery of the units has been delayed for over a month awaiting proper authorization for shipment. The units are being stored in a vault for safe keeping until authorization for shipment is received.

NAA Alclad Uranium-Aluminum Alloy Plates. During the quarter, work was initiated on an order for 330 Alclad uranium-aluminum alloy plates for physical mockup tests at North American Aviation, in connection with the Uranium-Converter Reactor. Some trouble is being experienced in manufacturing these high-uranium-content plates because of segregation during solidification of the alloy.

Nine-pound melts of 26 to 27 wt % uranium were prepared by the direct alloying method and cast into tilted graphite molds. Analysis of samples taken from the casting revealed a 1 to 1.5% variation in uranium content from top to bottom of the castings, as well as some side-to-side segregation. Tolerance on the uranium content in each core is stringent, that is, 8.79 ± 0.08 grams.

Despite the segregation problem, an attempt is being made to obtain alloy cores that are within the tolerance by using the density method. An experimental plot of density of the alloy as a function of composition has been obtained. This curve agrees well with the theoretical curve, except that it is slightly displaced. By determining the density of the individual cores, the uranium content in each core can be obtained from the curve.

Methods of reducing segregation in this alloy are being investigated. Such expedients as chill casting in cast iron, thinner cross section molds, and a 0.2% addition of titanium to the melt have failed to greatly reduce the amount of segregation encountered.

In preliminary tests, the possibility of preparing high-uranium-content plus aluminum compacts by powder metallurgy techniques appears to be promising. Cores of the MTR type, prepared by cold pressing a mixture of U_3O_8 and high-purity aluminum powder, show densities of the order of 90 to 95% of theoretical. Uranium oxide in the form of U_3O_8 is preferred because of its stability to the thermal treatment required during the subsequent jacketing and cladding operations that are done in air.

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