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REPORT TO THE WORKING COMMITTEE OF
THE FUEL ELEMENT DEVELOPMENT COMMITTEE

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REPORT TO THE WORKING COMMITTEE OF
THE FUEL ELEMENT DEVELOPMENT COMMITTEE

by:

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DOUGLAS UNITED NUCLEAR, INC.

September 29, 1966

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TABLE OF CONTENTS

Page No.

SMALL AND K REACTOR PRODUCTION FUELS

Uranium Core Production Data	3
Uranium Specifications	3
Fuel Performance	3
Non-Defense Plutonium.	5
Thoria Development	5
Uranium Alloy Testing.	5
Alternate Process Development.	8
Ultrasonic Vibration	8
Nickel Plating	14
Zirconium Plating.	15
Cladding Components.	15
New Cladding Development	16
Corrosion Program.	16

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SMALL AND K REACTOR PRODUCTION FUELS

Uranium Core Production Data

Inspection of Bare Cores

The combined visual and grain size reject rate of bare cores was 0.16 percent for the period April 1, 1966 through August 31, 1966.

Grain Size

The average grain size of salt-water and induction-oil heat treated cores received during the period March 3, 1966 through August 15, 1966 was between A-6 and A-7.

Uranium Specifications

Class II Cores

The uranium core specifications have been revised to include most of the proposed Class II categories described in the May 1966 Working Committee Report. The tightly-closed seam category is the only one not yet accepted.

Nitrogen Analysis

National Lead Company of Ohio has proposed eliminating the nitrogen analysis on each ingot as a cost-reduction item. The proposal is justified on the basis that the nitrogen rarely exceeds the specified limit without either the density being lower than the specified minimum or the magnesium limit being exceeded. A review of the analyses of 6,898 ingots showed 101 ingots that did not meet the specification for nitrogen of 100 ppm maximum, and 97 of these ingots had a density less than the specified minimum of 18.82 g/cc. The remaining four ingots all had a density greater than 18.82 g/cc but less than 18.91 g/cc. Rather than completely eliminating the nitrogen analysis, the core specifications will be revised to require nitrogen analyses only on those ingots with a density less than 18.91 g/cc. This will result in approximately ten percent of the ingots produced for DUN being analyzed for nitrogen content.

Fuel Performance

The fuel performance has continued to be good with seven fuel element failures occurring during the period April 28 through September 26, 1966. The failures are summarized in Table I. The cause of the hot-die-size overbore failure has not been determined. The failed element had a 1/2-inch diameter blister in the cladding in the vicinity of the downstream end of the downstream support, but there was no evidence of damage to the support nor was there any severe localized or uniform corrosion on the element. The two enriched hot-die-size fuel failures occurred in elements clad with HDA4 alloy (Al; 10% Si; 1% Ni; .5% Fe) which were being irradiated in a test to evaluate the corrosion resistance of HDA4 cladding.

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TABLE I

<u>Fuel Element Type</u>	<u>Reactor</u>	<u>Exposure (% of Goal)</u>	<u>Classification</u>	<u>Rupture Date</u>
Natural Non-Bumpered AlSi	KW	89	Side Hot Spot	4/30/66
Natural Non-Bumpered AlSi	D	77	Side Hot Spot	5/13/66
Natural - Overbore HDS	C	47	Side Unclassified	5/26/66
Natural Bumpered AlSi	C	49	Mechanical Damage	5/31/66
Enriched Bumpered HDS	D	54	Side Groove Corrosion	6/28/66
Enriched Bumpered HDS	D	76	Side Groove Corrosion	9/ 3/66
Natural Self-Support AlSi	KE	19	End Manufac- turing Defect	9/ 9/66

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Non-Defense Plutonium

Approximately 90 tons of depleted uranium (.14 wt. percent U-235) will be irradiated in the K Reactors to produce 215 Kg of plutonium containing 30 percent Pu-240. The irradiation will commence in January 1967 and will require approximately 18 months. The target average exposure for the depleted elements is 3600 MWD/T and the calculated maximum core temperature is 240 C. The specifications on the cores are essentially the same as for standard natural and enriched I&E cores, except that salt-oil heat treating has been specified. The cores have the same dimensions as the K5N and K4N models and will be AlSi canned.

Thoria Development

A test was conducted to evaluate the effect on closure welding of using a thoria wafer immediately below the end cap in thoria target elements. The test also evaluated the use of a heat-sinking chuck. The test conditions and results are summarized in Table II. The results indicate that good yields may be obtained by using a heat-sinking chuck, a thoria wafer against the end cap, or a combination of the two. The purpose of both is to prevent internal pressurization and blowback due to heat introduced to the core during welding. This is accomplished by rapid removal of heat through the side wall with the heat-sinking chuck or by the thermal insulation provided by the thoria wafer. The limitations of the two applied separately are that the heat-sinking chuck provides no control over heat flow through the cap-wafer into the core, and the insulating wafer provides no control over the temperature rise in the annular gap between the can and the wafer. Therefore, a combination of the two techniques should yield the best results.

Two groups of wafers were used in this test. Both groups had a thickness of 0.370 inch $\pm$ 0.022 inch and a density of 8.5 g/cc. One group had a diameter of 1.463 inch $\pm$ 0.011 inch, and the other had a nominal diameter of 1.490 inch. The aluminum cans used had a nominal inside diameter of 1.510 inch.

Uranium Alloy Testing

An irradiation test has been completed to determine the effects of iron, silicon, and aluminum and four different beta heat treatments on the dimensional stability of hot-die-size cores irradiated under current reactor operating conditions. The four heat treatments and the compositions tested are listed in Table III. The test was irradiated in the 15 central positions of 40 zirconium tubes in C-Reactor to an average exposure of 737 MWD/T.

National Lead Company of Ohio performed extensive X-ray diffraction studies on samples of the test material. The X-ray work consisted of measuring the texture longitudinally at the O.D., mid-wall, and I.D. on three cores from each test condition (52 total conditions). Four scans were made at each position on each core. The X-ray data and the irradiation-induced diameter changes have been correlated with each other, and the effects of iron, silicon, and aluminum on longitudinal G-3 and on diameter changes have been determined. The most significant and consistent alloy effect is that of iron on both the diameter changes and on the longitudinal G-3. Increasing iron consistently resulted in decreasing longitudinal G-3 at the O.D. of the cores and resulted

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TABLE II
THORIA TARGET ELEMENT CLOSURE
WELDING TEST

	Group					
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Number of Elements Welded	47	50	49	50	50	52
Conditions						
Heat-Sink Chuck				X	X	X
No Heat-Sink Chuck	X	X	X			
3/8" Gap Between Powder and Cap	X			X		
Powder Against Cap		X			X	
Thoria Wafer Against Cap			X			X
Percent Defective (Blowouts)	83	28	2	2	0	2

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TABLE III

CONDITIONS TESTED IN THE HOT-DIE-SIZE
CORE ALLOY TEST

<u>Composition</u>	<u>Constituent (ppm)</u>		
	<u>Fe</u>	<u>Si</u>	<u>Al</u>
1	175	300	300
2	175	50	300
3	175	300	50
4	175	50	50
5	300	175	300
6	50	175	300
7	300	175	50
8	50	175	50
9	300	300	175
10	50	300	175
11	300	50	175
12	50	50	175
13	175	175	175

<u>Heat Treatment</u>	<u>Heating Conditions</u>	<u>Transfer Time to Quench</u>	<u>Quench Conditions</u>	<u>Core Blank Form</u>
(1)	Immersion in 50% KCl-50% NaCl at 1350 F $\pm$ 10 F for 10-20 min.	20 $\pm$ 3 sec.	Water containing 5% salt at 95 F-130 F.	Solid - 1.485" O.D.
(2)	Immersion in 50% KCl-50% NaCl at 1350 F $\pm$ 10 F for 10-20 min.	20 $\pm$ 3 sec.	Water containing 5% salt at 95 F-130 F.	Hollow - 1.485" O.D. 0.411" I.D.
(3)	Immersion in 50% KCl-50% NaCl at 1350 F $\pm$ 10 F for 10-20 min.	10 $\pm$ 2 sec.	Hoghto-K oil at 115 F-150 F.	Hollow - 1.485" O.D. 0.411" I.D.
(4)	Induction heat to 1300 F-1385 F.	11 $\pm$ 1 sec.	Hoghto-K oil at 120 F-135 F	Hollow - 1.485" O.D. 0.411" I.D.

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in increasing diameter growth. The effects of silicon are neither so strong nor so significant, and aluminum has very little detectable effect. The results of the test are presented in Figures 1 through 5. Figure 1 shows the relationship between the average longitudinal G-3 and the average diameter change at the mid-point of the fuel elements for each test condition. The data show a significant relationship between G-3 and diameter change for each heat treatment, but that the relationships are not the same for all heat treatments. The graph shows the least-squares fits of the observed data points with the 95 percent confidence limits on the lines.

Figures 2 through 5 show the effects of iron and silicon on diameter changes and on the longitudinal growth index.

The water-quenched cores exhibited generally greater diameter growth than the oil-quenched cores. The induction-oil heat treatment resulted in slightly less diameter growth than the salt-oil for equivalent alloys.

Alternate Process Development

Ultrasonic Vibration

Further experiments were carried out in ultrasonic vibration of the fuel element caps during the hot-die-sizing operation. The objective of the work is to establish the feasibility of attaining bonds between the caps and the fuel core while the fuel is being die sized. The following process and component variables have been under study:

Sizing temperature:	350, 450, 550 C
Cap material:	8001, 10 w/o silicon-aluminum alloy
Vibration before sizing:	0 to 10 seconds
Cap thickness:	100 and 225 mils
Core end surfaces:	Smooth (not etched) Rough (normal etched) End grooves consisting of 90° including angle grooves 25, 50, and 100 mils deep

Evaluation thus far has included testing of the cap-core and base-core bonds by stud pulling. Temperature appeared to have a major effect, with a measurable bond strength only at the base end of fuels sized at 550 C. Values were inconsistent. Force required to pull a 0.19 inch diameter stud ranged from 300 to 725 pounds on four pieces tested, while four pieces in an earlier series of runs exhibited no bonding. The cap-core bond on one of these fuels ranged 400 to 425 pounds, while the remaining three were essentially zero strength.

Metallographic evaluation of cores having concentric V-grooves at the ends revealed diffusion bonding of 75 to 100 percent of the cap-core surface at

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Hollow Salt-Water Heat Treatment

FIGURE 3

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FIGURE 4

Effects of Iron and Silicon on
Irradiation-Induced Diameter Changes

Effects of Iron and Silicon on
Longitudinal Growth Index G-3

Hollow Induction-Oil Heat Treatment

FIGURE 5

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sizing temperatures of 450 C, and up to 30 percent of the surface bonded at temperatures of 350 C. Stud pull data are not in complete agreement with the metallographic observations with some values as low as 225 to 250 pounds force with 100 percent diffusion bonding indicated by metallography. However, on the group of fuels which appeared to be 75 to 100 percent bonded, stud pulls as high as 650 pounds were achieved.

Nickel Plating

A nickel fluoborate bath was employed in tests of electrodeposition of nickel on an aluminum substrate. In this bath, the major nickel salt is nickel sulfate, while approximately 1/3 of the nickel salt is fluoborate, and approximately 1/100 is nickel chloride. It has been described as being effective for plating directly on aluminum without an intermediate metallic or oxide layer.⁽¹⁾ Several specimens of annealed 8001 alloy were plated directly on aluminum with generally good adhesion as determined by bend testing and by thermal shock. The latter treatment consisted of a 20 minute residence time in a 525 C furnace followed by quenching in cold water. Metallographic evaluation revealed that this thermal shock treatment produced a visible Al-Ni diffusion layer approximately 0.05 mills thick, and continuous except for intermittent specks of unidentified dark material. A less continuous (approximately 50 percent visible) diffusion layer was produced by heat treatment for 16 hours at 400 C. Porosity of the plate was not tested.

Aluminum pretreatment for plating on bare aluminum consisted of a light caustic etch followed by desmutting in room temperature nitric acid, and finally a 10 to 15 seconds immersion in nominal 10 weight percent sulfuric acid at room temperature.

Samples of nickel plate produced by this bath were analyzed for boron content, including nickel foil and plated aluminum specimens: No significant boron contribution from the plating bath was indicated, with boron values generally at or near the lower limit of detection (20 ppm).

The fluoborate bath was also employed for test plating on 8001 alloy with an immersion-tin electrolytic-bronze coating. Adhesion was not satisfactory, as evidenced by gross blistering on removal from the plating bath.

It was discovered that with the processes employed, the aluminum alloy may markedly influence plate adhesion as evidenced by the blistering of 63-S alloys after pretreating, plating, and heat treating as described above.

Experiments in the electroplating of aluminum with nickel included testing the effects of post-plating heat treatment on nickel adhesion. Plated samples of annealed 8001 alloy aluminum were heat treated at 350 C for periods ranging from three hours to 100 hours, and at 410 C for 1/4 to sixteen hours. Following heat treatment, nickel adhesion was assessed by the "bubble machine" test (electrolytic generation of hydrogen on the plated sample as described in HW-71806, Appendix B). It appeared that 50 to 100

(1) Atkinson, J.T.N., "Novel Process for Plating on Aluminum Alloys." Plating, May 1960.

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hours at 350 C or five to ten hours at 410 C produced satisfactory adhesion, as indicated by absence of blisters on the nickel plate, following the "bubble machine" test. Metallographic studies are underway.

Zirconium Plating

A continuation of previously reported studies of the electrodeposition of zirconium on uranium included plating of both nickel-plated and electrolytically etched specimens, in an effort to improve the adhesion to uranium. A significant improvement was achieved in that there was no observable separation of the plate and substrate produced during cooling of the samples as occurred earlier. Approximately 50 percent of the sample surface had good adhesion, as indicated by probing. These samples were plated with one to two mils thick Zr at 700 C with a current density of approximately 15 amp/sq. ft. Further work was carried out at a bath temperature of 750 C with both nickel-plated and electrolytically-etched, unplated uranium. Adhesion was poor; however, these results are not considered conclusive since the plating bath was contaminated with both alumina and air.

Cladding Components

The inertia welding of aluminum wafers to aluminum tubes of varying wall thickness was tested in a study of cladding components fabrication techniques. Specimens prepared from 8001 aluminum alloy cap wafers and can components of varying wall thicknesses were used in the tests of inertia welding of the wafers to the end surfaces of the tube walls. The tube was held in a stationary position, and rotational energy was supplied the cap to produce heat for the weld by friction as the components were brought in contact. By this technique, flashing normally produced during the weld and the excess cap is machined away to expose the closure zone and the weld. Further, the weld appears radially on the lateral surface rather than on the end surface of the components.

After several unsuccessful attempts at joining the tube to a flat wafer, it was theorized that the wafer provided too great a sink for the heat produced and the geometry provided no space for metal flow. A projecting ring the same diameter and thickness of the tube was machined into the wafer. This also reduced wafer thickness, leaving a smaller heat sink. This wafer geometry was successfully inertia-welded to 0.085 inch thick tube. The weld zone of examined specimens is approximately 0.040 inch wide and exhibits excellent metallurgical structure which is sound and homogeneous. The weld is exposed on the lateral surface of the tubing.

Low heat generation capacity is believed to have prevented successful welds on thinner tubing.

Samples of 1245 aluminum alloy were included with the 8001 alloy samples. The 1245 alloy tubing was successfully joined to 8001 alloy wafers with less flashing produced from metal flow compared to 8001 tubing.

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New Cladding Development

On September 4, 1966, the irradiation test of hot-die-sized fuel clad in ten weight percent silicon-aluminum alloy was discharged from D Reactor when the second failure of the fuel in the test pattern occurred. The failures occurred at approximately 50 percent and 69 percent of the test goal exposure. The first ruptured fuel, in which water penetration occurred one to two inches from the downstream (male end) of the fuel, and at approximately 180° from the ribs, was located in column position 1 (downstream end). The cladding (including the bumper) was missing over an area approximating a 1 to 1-1/2 inch diameter circle. The top side of the fuel exhibited "ledge and groove" attack over an area 3/4 to 1 inch wide and extending to 1/2 inch from each end of the fuel. The usual brown film was absent over this area. Other fuels from the rupture column exhibited corrosion attack similar to the ruptured piece, but generally less severe, going upstream. Possible flow disturbance from bumpers appeared to be unrelated to the grooves.

The second failure occurred in piece number two (from downstream end) in a striped column of hot-die-size fuel clad in 8001 alloy and high silicon alloy. Preliminary examination of the failed element revealed an irregularly shaped penetration of the cladding less than one inch in diameter located about two inches from the upstream end of the fuel in the 12:00 to 3:00 o'clock quadrant. The failure was not associated with a bumper. A deep groove could be seen coming out of the center and a cluster of pits was located in the same quadrant toward the downstream end of the fuel. There was some evidence of ledge corrosion around the top supports.

Examination and measurement of the test material is under-way in C-Basin Measurements Facility. Limited preliminary data indicates weight losses of the high silicon-aluminum alloy-clad fuel to be greater than those of 8001 alloy-clad hot-die-size and 8001 alloy-clad AlSi process fuel by factors of about 1.2 and 1.4, respectively. Examination of the irradiated fuel revealed another fuel element whose cladding was penetrated by corrosion but which was not detected during the test.

Corrosion Program

Preparation of test fuels to evaluate the corrosion resistance of dichromate autoclave films in K-Reacto is nearing completion. The autoclaving consists of two 25 to 30 hour cycles, first in deionized water followed by a cycle in eight to ten weight percent potassium dichromate. A major process variable is autoclaving temperature, and the following values are employed for this test:

<u>Fuel Type</u>	<u>D.I. Water Temperature, °C</u>	<u>Dichromate Temperature, °C</u>
A	170	170
B	290	170
C	290	230
D	340	230

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Controls are autoclaved in 170 C process water for 50 hours. The fuel is being prepared for irradiation under PTA-026.

Corrosion testing under high flow velocities was terminated. No reproducible results were obtained. There were indications, however, that the aluminum jacket will pit-corrode if it is anodic to the surrounding tube.

Corrosion tests in ex-reactor process tubes using heated reactor process water are not yet conclusive. Neither the pieces being run as TF-18 Test 1B (after 4-1/2 months) nor the autoclaved versus non-autoclaved pieces (after six weeks) showed any significant corrosion.

Tests exploring the effects of ultrasonic vibration on corrosion have been terminated. Preliminary evaluation of results indicate:

- a) Cavitation produced by ultrasonics increases the frequency of groove formation by two to three fold.
- b) Comparing autoclaved and non-autoclaved pieces, the autoclaved pieces exhibit approximately 50 percent more grooves than the non-autoclaved.
- c) The high-silicon (10 w/o silicon-aluminum) alloy is more resistant than X-8001 or C-64 to both uniform corrosion and to cavitation grooving. Compared to the high silicon alloy, the groove frequency of C-64 increases by approximately two-fold, and 8001 by approximately three-fold.

The effect of reversing the current flow from that found by natural galvanic action was studied in the corrosion flow test equipment. The galvanic voltage was measured in a K-Reactor process water system, and twice that amount was applied to the system in reverse polarity. The test period was 24 hours. In a test period of 24 hours, the X-8001 showed numerous pits covering over 50 percent of the test area; however, there was very little uniform corrosion. If the total amount of aluminum removed as pits were spread over the entire surface, approximately four mils would have been removed. A repeat test confirmed these results.

A preliminary study of the AlSi braze layer was initiated to evaluate the corrosion behavior of the braze. Radiometallurgy examinations of irradiated fuels have shown some evidence of higher resistance of the braze layer to penetration by the reactor cooling water. Samples taken incrementally through the braze layer have been subjected to corrosion environment to measure penetration rate. The corrosion tests are being made at 80 C in a solution of sodium acetate, sodium chloride, and acetic acid buffered to pH 4.3. Rate of penetration of corrosion is measured in selected corrosion pits. Initial data from the tests indicate a significant decrease in the rate of penetration as the corroding surface approaches the uranium-AlSi interface. Tests and analysis of the data are continuing.

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