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DOUGLAS UNITED NUCLEAR, INC.

REPORT TO THE WORKING COMMITTEE OF

THE FUEL ELEMENT DEVELOPMENT COMMITTEE

by:

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SMALL AND K REACTOR PRODUCTION FUELS

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Uranium Core Production Data

Inspection of Virgin Cores

The combined UT-2 and visual bare core reject rate was 0.30 per cent for the period September 1 through November 30, 1965.

Grain Size

The average grain size of cores received during the period July 1, 1965 through October 15, 1965 was between A-5 and A-7 on salt-water heat-treated cores, and between A-4 and A-5 on salt-oil heat treated cores.

Uranium Specifications

Changes in the FMPC Manufacturing Specifications were approved in October. The principle changes permit remelted top crops to be considered as solid remelt metal rather than as a separate item in the ingot charge composition and permit the use of one-derby charges when there is not sufficient virgin metal for continuous two-derby charges.

Fuel Performance

Eight normal and six enriched AlSi-brazed fuel elements failed during the period September 1 through December 15, 1965. These failures are summarized in Table I. The two elements that failed in D Reactor October 13 and October 25 were part of a run-to-rupture test comparing hot-die-sized and AlSi-brazed fuel elements, which is discussed in the section on alternate process development. Of the other 12 failures, five were caused by side hot spots in non-bumpered fuel and two were caused by end manufacturing defects, probably brittle end bonds, in K Reactor self-supported fuel. Both types of failures should be virtually eliminated by plans to use bumpers on all non self-supported fuel models in the winter months beginning the winter of 1966 - 1967 and by a change in the silicon content of the duplex bath to eliminate brittle end bonds.

The external bond count trends are shown in Figure 1. Sufficient post-irradiation data is not available to extend the work trend beyond that shown for the last reporting period.

Depleted Uranium

The depleted uranium load of approximately 12.5 tons was discharged from B Reactor on November 2, 1965 at an average exposure of 2475 MWD/T, and the ten ton load was discharged from KE Reactor on October 21, 1965 at an average exposure of 2700 MWD/T. These discharges include eleven monitor columns from KE Reactor and five from B Reactor, but the examination of the monitor columns is not yet complete. One column of depleted elements has been left in each of the two reactors for irradiation to 4000 MWD/T.

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TABLE IALSi Fuel Element Failures

<u>Fuel Element Type</u>	<u>Reactor</u>	<u>Per Cent of Goal Exposure</u>	<u>Rupture Classification</u>	<u>Rupture Date</u>
Enriched Non-Bumpered	C	75	Side Hot Spot	9/26/65
Natural Non-Bumpered	B	79	Side Unclas- sified	9/30/65
Enriched Non-Bumpered	B	89	Hole Manufac- turing Defect	10/13/65
Enriched Non-Bumpered	D	58	Side Groove Corrosion	10/18/65
Enriched Non-Bumpered	D	69	Side Corrosion	10/25/65
Natural Self-Support	KE	53	End Manufac- turing Defect	10/26/65
Natural Self-Support	KE	78	End Manufac- turing Defect	11/11/65
Natural Non-Bumpered	KW	75	Not Examined	11/14/65
Natural Watermix	B	168	Side Groove Corrosion	11/18/65
Natural Non-Bumpered	KE	91	Side Unclas- sified	11/21/65
Natural Non-Bumpered	C	44	Side Hot Spot	11/22/65
Enriched Non-Bumpered	C	60	Side Hot Spot	11/26/65
Enriched Non-Bumpered	C	54	Side Hot Spot	12/ 2/65
Natural Non-Bumpered	KW	92	Side Hot Spot	12/ 5/65

FIGURE 1

BM-5300-074(3-60)
AC 1.2 200000 70000

FUEL ELEMENT EXTERNAL BOND COUNT

AVERAGE LOG BOND COUNT - $\text{LOG}(x + 1)$

AlSi Process Development

A large-scale canning test is being conducted in an effort to eliminate the brittle end bonding on the K Reactor self-support models. Initial tests showed that a duplex silicon content of seven per cent improved the end bonding but caused excessive external lateral bond rejects due to the formation of a sludge in the bond layer, and that the optimum silicon content is probably around eight per cent. Testing is now in progress with the duplex furnace operating at a nominal eight per cent silicon on two lines and at the standard nine per cent silicon on one line. After eight days of testing, the control line showed a ten per cent external bond reject rate and the test lines a 1.2 per cent external bond reject rate. Samples of the test and control elements are being evaluated by metallography.

Thoria Program

A third thoria canning campaign began in October, and approximately 25,000 elements for charging in the cores of the K Reactors have been made. The thoria powder for this campaign has been produced by Mallinckrodt Chemical Works, and the quality of the powder is substantially higher than that used in previous runs. Compaction densities of 7.1 to 7.5 g/cc have been easily achieved, and all of the thoria has met the specification on uranium of ten ppm maximum and the LOI specification of 0.10 per cent maximum. The principle problem during canning continues to be the rejection of elements for defective welds. The over-all yield is around 85 per cent with the largest reject categories being blowouts, non-covers, improper core strikes, and voids found in radiograph inspection. Only 0.27 per cent of the elements have been rejected by the helium leak test.

Two thoria elements have failed during irradiation since September, bringing the total number of thoria failures to 23. Both elements were canned in .090 inch thick components, and both failures occurred in the fringe of KE Reactor, one on September 13, 1965 after 34 days of irradiation, and the other on October 21, 1965 after 72 days of irradiation.

Alternate Process DevelopmentFuel Performance

Two hot-die-size clad fuel elements failed during the reporting period. The failures are summarized in Table II.

TABLE IIHOT-DIE-SIZE FUEL RUPTURES

<u>Fuel Element Type</u>	<u>Reactor</u>	<u>Per Cent of Goal Exposure</u>	<u>Rupture Classification</u>	<u>Rupture Date</u>
Natural Over- bore	C	82	Side Unclas- sified	9/22/65
Enriched Non- Bumpered	D	58	Side Groove Corrosion	10/10/65

The cause of the overbore failure has not been precisely determined. It may have been either water entry through the closure weld or side corrosion. The enriched failure occurred in the run-to-rupture test which is described later in this section of the report.

Ledge and Groove Corrosion

Localized corrosion of aluminum-clad fuel elements, defined as ledge and groove corrosion, is routinely observed on some elements irradiated in the downstream fuel positions of all Hanford production reactors. The incidence of groove corrosion has not been consistent; however, there have been strong indications that alum feed rate to the reactor coolant has contributed to an increase in groove corrosion attack. The frequency of groove corrosion on K Reactor fuel elements decreased during a period when the pH of the coolant was lowered from 7.0 to 6.6. Audit of fuel performance under the Quality Certification Program has reflected an annual cyclic pattern to groove corrosion in which the frequency is higher in the period from August through October.

Recent irradiation experience indicates that the groove corrosion attack of hot-die-sized fuel may be somewhat more severe and more frequent than that observed on the AlSi-brazed fuel. Figure 2 shows irradiated AlSi-brazed and hot-die-sized fuels exhibiting typical groove corrosion attack patterns.

During July 1965, an irradiation test, PT-IP-781-A, was charged in D Reactor to compare the irradiation behavior of hot-die-sized fuel and AlSi-brazed fuel. The test, consisting of twelve columns each of matched enriched hot-die-sized fuel and AlSi-brazed fuel, was designed to provide a statistical comparison of fuel performance by irradiation of the test material until a planned number of fuel failures occurred. The statistical design of the test was based upon a mathematical rupture model which defines the rupture rate due to process-related causes. Fuel failures in the test occurred in the following sequence:

<u>Date</u>	<u>Fuel Process</u>	<u>Exposure (MWD/T)</u>	<u>Cause of Failure</u>
8/29/65	HDS	410	Groove Corrosion-Side
9/ 7/65	HDS	450	" "
10/10/65	HDS	~ 850	" "
10/18/65	AlSi	~ 900	" "
~10/22/65	AlSi	~1000	Side blowout. Point of coolant entry not known. Severely grooved at location of failure.

Within the test pattern of 24 columns of fuel there were six new aluminum reactor tubes. All the fuel failures occurred in five of the six new reactor tubes in the test.

FIGURE 2

AlSi Fuel Element Showing Typical Groove and Pitting Corrosion Attack. The Vertical Projections in the Lower Part of Picture are Self Supports.

HDS Fuel Element from PT-781 Showing Typical Groove and Pitting Corrosion Attack.

As the failures occurred in the test, the fuel elements in the discharged columns were examined. The first ten to eleven elements from the downstream end of the fuel columns exhibited a high frequency of groove corrosion. Preliminary test data indicated that the groove corrosion behavior of hot-die-sized fuel and AlSi fuel may have been different, since AlSi fuel discharged from columns close to the test pattern exhibited a lower frequency than the hot-die-sized test fuel. Subsequent examination and measurements of the test showed:

Number of Elements	Type	Per Cent of Fuels Exhibiting Groove Corrosion				Total
		< 0.010 In.	0.010 - 0.020 In.	0.020 - BL(*) In.	BL(*)	
390	AlSi	8.8	12.1	1.5	9.2	31.5
403	HDS	19.9	13.7	3.7	---	37.5

* BL indicates braze layer or nickel plate exposed.

The test material exhibited a higher frequency and greater severity of groove corrosion on the upper half of the fuel elements as they were positioned horizontally in the reactor tubes. The corrosion was more frequent toward the ends of the fuels, particularly the cap ends, which are oriented downstream in the reactor tubes. Variations in grain size of the aluminum cladding appeared to have little or no effect on frequency and severity of groove corrosion. Pre- and post-irradiation measurements data are undergoing analysis and evaluation to determine the effects of irradiation and fuels variables on the corrosion behavior of the fuels. Raw data as yet not analyzed indicate the weight loss of AlSi-brazed fuel to be greater than that of hot-die-sized fuel by a factor approaching two.

When the initial data from PT-781-A showed evidence of differences in groove corrosion behavior between the AlSi and hot-die-sized fuel, a program was established for examination of hot-die-sized fuel which was being routinely irradiated in K Reactor. Examination of the downstream fuels from K Reactor revealed:

Reactor	Goal Exposure	Fuel Elements Examined		Per Cent of Fuels Exhibiting Groove Corrosion		Maximum Depth of Corrosion (Est.), Mils	
		HDS	AlSi	HDS	AlSi	HDS	AlSi
KE	50	158	152	25	13	20	< 10
KW	100	380	190	36	4	20	< 10

A review of data from all previous hot-die-sized fuel production tests shows:

PT	Per Cent of Fuels in Downstream Positions Exhibiting Groove Corrosion			
	Greater than 10 Mils		Less than 10 Mils	
	HDS	AlSi	HDS	AlSi
546	3.6	2.0	13.1	13.2
616	---	---	2.0	1.3
665	---	---	5.7	35.2

PT	Per Cent of Fuels in Downstream Positions Exhibiting Groove Corrosion			
	Greater than 10 Mils		Less than 10 Mils	
	HDS	AlSi	HDS	AlSi
546A	0.8	---	2.5	7.5
684	---	---	10.9	16.3
708(*)	6.5	13.9	6.6	81.9
677	1.9	---	16.3	19.4

(*) Downstream positions of PT-708 contained AlSi fuel in positions 1-4 and HDS fuel in positions 5-16.

All differences in the above table except the less than 10 mil category of PT-546 are statistically significant. The corrosion behavior of all the test fuel, however, may have been influenced by the higher than normal dimensional changes of the water-quenched uranium core fuel in all the production tests. In addition, the major portion of the fuel columns contained hot-die-sized and AlSi fuels in alternate positions in the columns. The influence of such a charge pattern on corrosion behavior is not known, but could be significant.

Irradiation behavior data compiled thus far has not been analyzed and evaluated. Conclusions and findings are therefore subject to confirmation by statistical analysis of the data. Initial data strongly pointed to a marked difference in groove corrosion behavior between hot-die-sized fuel and AlSi-brazed fuel. More rigid evaluation of the data and additional data compiled in the closely controlled irradiation test PT-781-A tends to refute the initial findings. The existence of an actual difference in behavior between hot-die-sized and AlSi-brazed fuel has not as yet been confirmed.

The development program directed toward resolution of the groove corrosion problem included studies in a number of areas involving fuel properties and behavior. In an effort to exploit any differences in corrosion behavior between hot-die-sized and AlSi-brazed fuel, a study was undertaken to determine metallurgical differences in the cladding of the two processes. Specific properties of the fuels under study are cladding grain size and structure, hardness, residual stress, intermetallic phase dispersion, chemical composition, and resistance to corrosion. A study of the influence of the hot-die-sizing process on fuel cladding includes the effects of preheating and sizing, end bonding, and aluminum component pretreatment.

The macro-structure of hot-die-sized and AlSi fuels cladding were compared, and on the basis of grain size, the hot-die-sized fuels were found to be less uniform than the AlSi. Typical elements of the groups etched are shown in Figure 3. Metallographic examination of hot-die-sized fuels revealed:

- (1) Fine-grain structure from the male end (cap) to approximately 0.4 inch from the male end.

FIGURE 3

Typical AlSi (Left) and HDS (Right)
Fuel Elements, Etched to Show
Typical Grain Structure of each
Type Element.

- (2) Large grains (up to approximately 0.050 inch diameter) from 0.4 inch to approximately 1-3/4 inches from the male end.
- (3) Fine grains in an approximate three-inch long center section.
- (4) Large grains from approximately 1-3/4 inches to approximately 0.3 inch from the base end (but finer than those near the cap end).
- (5) Fine grains from the base end up to approximately 0.3 inch from the base end.

Examination of micro-structure of the cladding of each element type confirms the inspection and analysis of the macro-structure. The elements were examined at three locations in directions longitudinal, transverse, and normal to the axis of the element. The grain size and structure were similar at each location and direction in the cladding of the AlSi element (see Figure 4). The grain size and structure of the hot-die-sized elements were similar in all directions at each location; however, the grain sizes were much larger at the ends of the hot-die-sized elements. Some large grains also appeared in the mid-section of the cladding that were not evident in the macro-examination. The longitudinal mid-point grain size and structure gave evidence of additional cold working that occurs in the sizing operation.

Hardness test results have been inconsistent, but the latest measurements indicate that the hot-die-sized and AlSi-brazed fuel claddings have the same average hardness. Greater variation in cladding hardness was found in the hot-die-sized fuel. A comparison of hardness values is shown in Figure 5.

Samples of cladding from a single hot-die-sized fuel element indicate that the cladding is under compressive stress. Additional samples of other hot-die-sized fuels are being examined for confirmation of these findings. Similar tests are scheduled to be made on AlSi-brazed elements for comparison.

The dispersion and size of intermetallic phases in the aluminum cladding exhibited no difference in unirradiated hot-die-sized and AlSi-brazed fuel elements. Radiometallurgical examination of an irradiated hot-die-sized fuel element revealed no differences in dispersion and size of intermetallic phases either under heavily corroded or non-corroded areas of the cladding.

Spectrographic analysis of the aluminum cladding indicated that the samples of hot-die-sized cladding contained 0.06 to 0.16 per cent copper as compared with 0.02 per cent copper in the AlSi-brazed fuel cladding. These amounts are within the specified limits for copper contamination. Differences are attributed to the fact that the two components were obtained from different vendors.

Examination of the cladding-core bond layer of irradiated fuel elements in the Radiometallurgy Laboratory has shown no evidence of bond deterioration, even under areas of severe groove corrosion attack. High bond

HDS

FIGURE 4

AlSi

1/4 to 1/2
inch from
Cap End

Midpoint

1/2 to 1
inch from
Base End

Aluminum Grain Structure of HDS and AlSi Fuel Element Cladding
Transverse to the Surface at Three Locations.

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strength was demonstrated by small portions of uranium core which remained bonded to the aluminum cladding of a failed element.

Corrosion tests are being conducted in testing equipment designed and fabricated to simulate groove corrosion. The test facility employs high velocity water under conditions which permit varying temperatures, velocity, and composition of the water. The objective of the tests is to permit a comparison of corrosion behavior of different areas of the hot-die-sized fuel cladding with that of the AlSi-brazed fuel cladding. An attempt will be made to correlate test corrosion behavior with irradiation behavior of the fuel. Using process water at approximately 100°C with a velocity of 80 ft./second, grooves have been produced in the cladding of hot-die-sized fuel having similar severity to those observed on irradiated fuels. Preliminary data indicates differences in corrosion behavior at different locations on the hot-die-sized fuel cladding.

Hot-die-sizing process variable tests have included preheating, sizing, and end bonding. Some annealing of the aluminum component was found to take place during preheating of the assembly; however, the die sizing operation reworks the aluminum to a uniform grain structure. The non-uniformity of structure exhibited in Figure 3 is therefore attributed to the heat and pressure applied to the fuel element during the end bonding operation. No significant effect was observed on the cladding by varying wall reduction ratio in the die sizing operation. In one sample of components, annealing of the components prior to die sizing caused a marked increase in grain growth of the cladding. This behavior has not as yet been confirmed in other components. Preliminary data indicate that the cladding fuels fabricated from aluminum tubing components experiences less grain growth than that of fuels fabricated from impact-extruded components.

The irradiation of hot-die-sized fuel is scheduled to continue under closely controlled conditions. Continuous charging of hot-die-sized fuel monitor columns will provide a routine audit of the corrosion behavior of the fuels in all reactors. Selected routine charging of hot-die-sized fuel will provide irradiation experience which will permit continuation of the fuels fabrication evaluation. Tests of alternate aluminum alloys and autoclave films are planned to determine the effects on corrosion behavior. A preliminary irradiation test of enriched fuels clad in 10 per cent silicon-aluminum alloy is being prepared for charging in a small reactor. Irradiation tests planned in the immediate future include an evaluation of the effects of fuels process variables on corrosion behavior and the resistance to chromate-dichromate and other films to corrosion during irradiation.

Core Optimization

A test is being prepared for reactor charging to determine the optimum core composition and heat treatment for hot-die-sized fuel and current reactor operating conditions. Thirteen different alloys containing iron, silicon, and aluminum at 50, 175, and 300 ppm and four heat treatments; solid salt-water, hollow salt-water, hollow salt-oil, and hollow induction-oil are being evaluated. The solid salt-water heat treated cores of the

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300 ppm Fe - 300 ppm Si - 175 ppm Al alloy had severe circumferential cracking which is shown in Figure 6. The hollow salt-water heat-treated cores of the same composition were not cracked, nor were the solid heat-treated cores of other compositions. The compositions included in this test are shown in Table III.

TABLE III
CORE OPTIMIZATION TEST
ALLOY COMPOSITIONS (ppm)

<u>Iron</u>	<u>Silicon</u>	<u>Aluminum</u>
175	300	300
175	50	300
175	300	50
175	50	50
300	175	300
50	175	300
300	175	50
50	175	50
300	300	175
50	300	175
300	50	175
50	50	175
175	175	175

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Improved Nickel Plating

Research work on the nickel plating of uranium has been primarily directed toward producing a coating which resists penetration by water for an extended period of time, and thus may serve as a secondary corrosion barrier in the event the aluminum jacket fails. Initial tests include the alteration of uranium pretreatment and plating conditions to reduce plate porosity. Specifically:

- (1) An electrolytic etch solution was developed which provides a smoother uranium surface than the standard etch, and as a corrolary advantage removes less uranium from the surface. The improved etch is an aqueous bath of 0.32 M HCl, and 6.0 M H_3PO_4 . (The standard etch is 0.35 M HCl, 6.0 M H_3PO_4 .)
- (2) Bromide ion was substituted for chloride ion in the Watts plating bath. It was found that the plating bath was much less corrosive to uranium, and anode efficiency was not impaired.

Employing these improved techniques, it was found that a 0.4 mil thick nickel plate gave protection equivalent to a 0.7 mil thick plate produced using the standard process (H_3PO_4 -HCl etch and chloride plating bath) as indicated by heating plated specimens in air.

FIGURE 6

Cracked C5AN Cores
Salt-Water Heat Treated
300 ppm Fe - 300 ppm Si - 175 ppm Al

Recent work has included an evaluation of the resistance of nickel-plated uranium specimens to attack by 170°C saturated steam.

Both the rough etch (H_3PO_4-HCl) and the smooth etch (H_2SO_4-HCl) were employed with nickel plates one and two mils thick. Based on behavior of one or two samples of each type, the smooth etch having a two mil thick plate resists penetration for 30-33 hours, compared to six to ten hours for the rough etch. Both types of etch with a one-mil thick plate failed in two hours or less. Application of additional coatings is to be investigated as a means of improving resistance to water penetration. Hydrogen-resistant materials are being considered.

Another method of possibly improving nickel-plate integrity is use of pulsed plating current. Employing a pulse at high current density, followed by an interval of zero current density, chemical changes in the liquid layer adjacent to the cathode may be minimized. Nickel plates ranging from 2 to 2.5 mils thick were produced employing current "ON" times ranging from 100 to 2000 micro-seconds, followed by "OFF" times ranging from 800 to 2000 micro-seconds. Plates satisfactory in appearance were produced, with plating current densities up to 1000 amp/sq. ft. Metallographic study of the samples is underway at present.

Zirconium Plating

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Zirconium has been successfully electro-deposited from a fused salt bath, and laboratory-bench scale experiments are underway to improve the coating. The bath consists of a Li-NA-K-fluoride eutectic with a melting point of approximately 450°C. Zirconium is dissolved as zirconium tetrafluoride and the Zr^{+4} ion is continuously replaced by using a zirconium anode.

The container is a nickel-lined, covered cell, externally heated, equipped with argon gas blanketing and agitation. Results to date are very preliminary, and the zirconium has been deposited on a copper cathode as a means of simplifying the initial experiments. It was found that addition of 10 weight per cent ZrF_4 to the electrolyte increased the melting point by 50 to 100°C, and thus a temperature of 600°C was used for plating. At current densities of 10 and 100 amp/sq. ft., a one-mil thick, fine-grained, dense plate was produced, as indicated by visual examination, but the plate became very nodular as the thickness was increased. Adherence was good as determined by sectioning and abrading the samples. Further experiments will be directed toward achieving a lower viscosity bath in the range of 550 to 600°C, and in further investigation of bath operating conditions.

Nickel Thickness Tested

Investigation of several devices for nondestructively measuring the thickness of nickel plate has established three devices which are capable of measuring by continuous scanning or by spot-readings, and either on bare nickel (as-plated on uranium) or through the aluminum cladding on a finished fuel element. Accuracy is about ± 0.1 mils in the range of 0.3 to 1.0 mils total thickness. Data are presented below in Table IV for the following instruments:

- #1 The present device, suitable only for bare Ni on U.
- #2 A magnetic device measuring deflection of a needle, actuated by the attractive force of the nickel plate on a permanent magnet, suitable only for spot checking, not continuous scanning.
- #3 A "nickel detector" employing the magnetic properties of nickel and effects in an electronic circuit.
- #4 A "Hall Effect" device.

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TABLE IV

NICKEL THICKNESS TESTER PERFORMANCE

		Ni Thickness, Mils		
		0.31(1)	0.69(1)	1.09(1)
#1	Reading	85	70	56
On Bare Ni	Standard Deviation	3	3	3
#2	Reading	24	35	53
On Bare Ni	Standard Deviation	1	1	1
#2	Reading	30	45	65
With Al over Ni(2)	Standard Deviation	4	4	4
#3	Reading	26	36	54
On Bare Ni	Standard Deviation	4	4	4
#3	Reading	16	31	69
With Al over Ni(2)	Standard Deviation	2	2	3
#4	Reading	16	22	32
On Bare Ni	Standard Deviation	2	2	1
#4	Reading	25	34	43
With Al over Ni(1)	Standard Deviation	1	2	2

(1) By metallographic measurement.

(2) Nominal 30-mil thick jacketing applied by the hot-die-sizing process.

Both #3 and #4 are suitable for continuous scanning, and the precision of the measurement is about the same for both through 30-mil thick aluminum. #4 is being tested in pilot plant work since it has better precision over bare Ni, and its output signal was considerably stronger than that of #3, indicating it is capable of measuring through thicker aluminum.

Standard deviations are not highly accurate, since they were interpolated from a few plotted points. However, the values are satisfactory for comparing instruments.

Weld Testing

Experiments are under way to detect pinhole defects in fuel element closure welds by spectrophotometric monitoring of the welding arc. Feasibility tests showed that welding arc light could be transmitted to a spectrometer using lenses and a "light pipe", and that the equipment was capable of detecting impurities deliberately added to the weld zone, including oil and water, producing a graphical readout. Improved equipment having higher resolution powers has been obtained, and is being employed to monitor the production welding operation on a test basis. If hydrogen and other contaminants can be detected, an attempt will be made to correlate the spectrophotometric evaluation with the weld defects as determined by visual inspection.

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