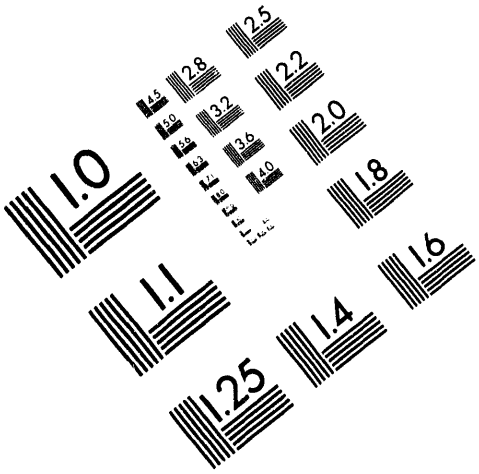

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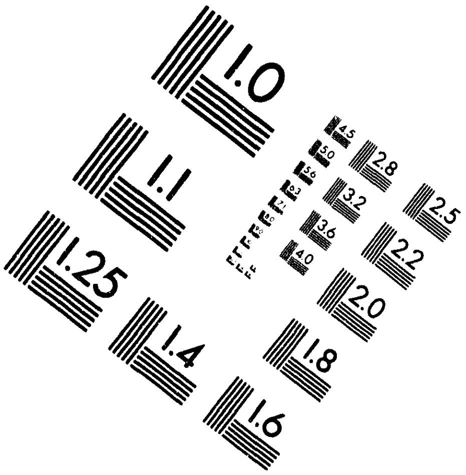

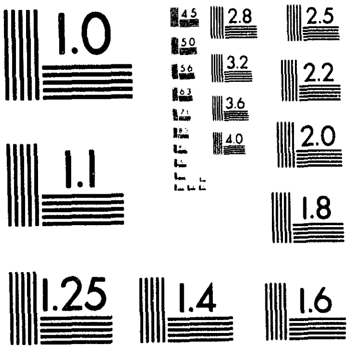

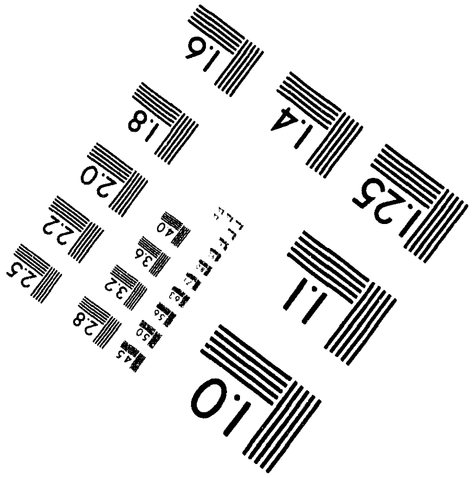

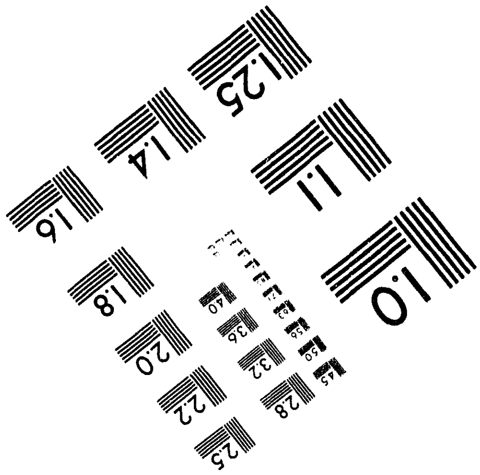

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TITLE

FABRICATION OF HOT DIE SIZED DIFFUSION BONDED FUEL ELEMENTS FROM OIL AND WATER QUENCHED CORES FOR PRODUCTION TEST IP-708-A

AUTHOR

C. A. STRAND

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FABRICATION OF HOT DIE SIZED DIFFUSION BONDED FUEL ELEMENTS FROM OIL AND WATER QUENCHED CORES FOR PRODUCTION TEST IP-708-A

September 15, 1964

By:

C. A. STRAND
Process Development Unit
Fuels Engineering Operation
Production Fuels Section
IRRADIATION PROCESSING DEPARTMENT

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FABRICATION OF HOT DIE SIZED DIFFUSION BONDED FUEL ELEMENTS FROM OIL AND WATER QUENCHED CORES FOR PRODUCTION TEST IP-708-A

INTRODUCTION

The hot die sizing (HDS) process is being considered as a replacement for the existing AlSi brazing process. Fuel cores are clad by passing a preheated core-component assembly through a die to bond the outside surface. Concurrently, the assembly is passed over a die plug to bond the internal surface. End bonding is accomplished in a following step by applying heat and pressure to the sized fuel element. Remaining fabrication steps are essentially the same as those used for AlSi brazed fuel.

Initial irradiation tests indicated that hot die sized fuels experienced in-reactor growth behavior which differed from that of the matched AlSi fuels in the tests.

Presently, two tests are being irradiated in reactor for determining the effect of end-bonding method, and end-bonding pressure in combination with fuel core geometry, upon fuel element diametral growth during irradiation. Due to the length of time required for irradiation and examination, a third test to determine the effect of core heat treatment and stress relieving has been prepared and is ready for reactor charging. This report summarizes the fabrication of oil and water-quenched cores pretreated by both alpha annealing and AlSi brazing for "Production Test IP-708-A, Evaluation of Oil Quenched Hot Die Sized Elements," HW-83454.

SUMMARY

Eighteen weighed and measured charges of hot die-sized fuel elements have been prepared and are ready for delivery to C Reactor. The charges consist of twenty-four test pieces in the mid-section of each charge. AlSi brazed fuel has been used as filler material in the first and last four positions of each tube. The test section consists of a statistical arrangement of six different groups of water and oil-quenched material. Each type contained alpha annealed, AlSi braze pretreatment, and standard as-received cores to test the effect of stress relieving.

Although the nondestructive testers indicated the existence of a bonding problem early in fabrication of the test pieces, it was subsequently established that no process problem existed. Fabrication of the test pieces was smooth and with the exception of the above-mentioned suspected problem, no difficulties were encountered. Seventy-nine percent of the pieces sized were acceptable for autoclaving.

DISCUSSION

Six-hundred and thirty fuel elements were sized and finished during the period July 13-31, 1964, for continued irradiation testing of hot die sized fuel. The uranium cores for this test consisted of matched oil-quenched and water-quenched pieces taken from alternate positions within each rod of core stock material. Each of the two types of material were divided into the following three groups for the purpose of testing the effect of stress relieving:

Group 1 - No treatment
Group 2 - Alpha annealed
Group 3 - AlSi brazing pretreatment

A yield of 79 percent of the pieces sized was achieved for this run. Yield and reject rates were based upon pieces sized to show process capabilities. Station reject rates are shown in Table 1. A large number of cores (56) were not sized due to a shutdown (July 15) to determine the extent of a suspected bonding problem. When it was determined that no problem existed, the run was resumed on July 20. No unusual difficulties were observed in this run with the exception of the delay caused by faulty operation of the non-destructive testers. The fully automatic induction heated end-bonding presses performed well. Sizing yield was a little low as reflected in the reject rate of 9.5 percent. Part of the rejects were caused by an alignment and set-up problem as well as a slight core distortion due to the pretreatments. Alpha annealing and AlSi pretreatment caused some core warpage, which is believed to have contributed to the sizing rejects.

Data taken during processing is recorded in the following notebooks:

Nickel Plating - HW-81229
HDS Process Data - HW-83454A

Processing of the various core types will be discussed in detail under each of the process steps in the order of processing as shown by flowsheets in Figures 1 and 2.

TABLE 1
TABULATION OF STATION REJECT RATES

Station	Number to Station	Station Rejects	
		No.	Percentage
Nickel Plating	788[1]	102	12.94
(Cracks)		(45)	(5.71)
(Contact Burns)		(47)	(5.96)
(Other - Stains, Oxide, etc.)		(10)	(1.27)
Sizing Total	630	60	9.52
(Wrinkled)		(36)	(5.71)
(Pulled Spire)		(14)	(2.20)
(Off Cycle)		(10)	(1.60)
Trim	570	4	0.70
End Bond	566	5	0.88
Ream	561	10[2]	1.78[2]
Inner Bond (UE-1)	551	1	0.18
Outer Bond (UE-1)	551	2	0.36
Inner Closure (UT-4)	548	2	0.36
Outer Closure (UT-4)	548	8	1.46
Inner Clad (UE-3)	538	0	0
Outer Clad (UE-3)	538	0	0
Cap Bond (UE-3)	538	14[3]	2.60
Base Bond (UE-3)	538	8[4]	1.49
Etch	516	0	0
Facing	516	0	0
Inner Weld	516	8	1.55
Outer Weld	516	7	1.36
Inner Reweld	501	0	0
Outer Reweld	501	1	0.20
Rail Weld	500	1	0.20
Marred Surface	499	1	0.20

Yield to autoclave (percentage of pieces sized): 498 pieces - 79.05%

[1] 56 pieces were not sized when run stopped to examine supposed bonding problem.

[2] 10 pieces taken at ream station for destructive examination. No pieces rejected for defects.

[3] 10 actual rejects; 4 false rejects.

[4] 4 actual rejects; 4 false rejects.

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FIGURE 1

OIL AND WATER QUENCHED CORE PREPARATION

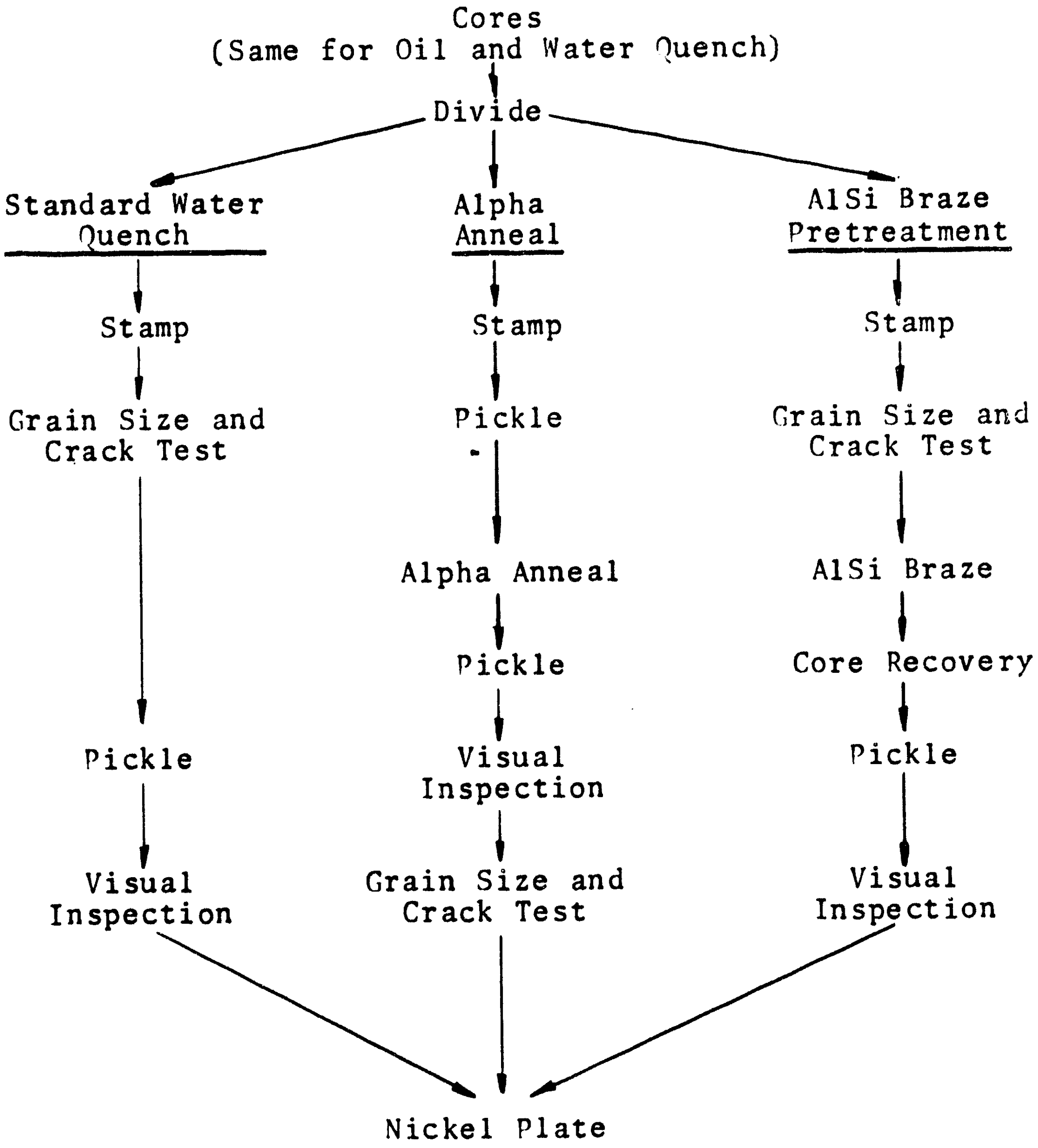

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FIGURE 2
HDS PROCESS FLOW SHEET FOR OIL AND WATER QUENCHED CORE FABRICATION

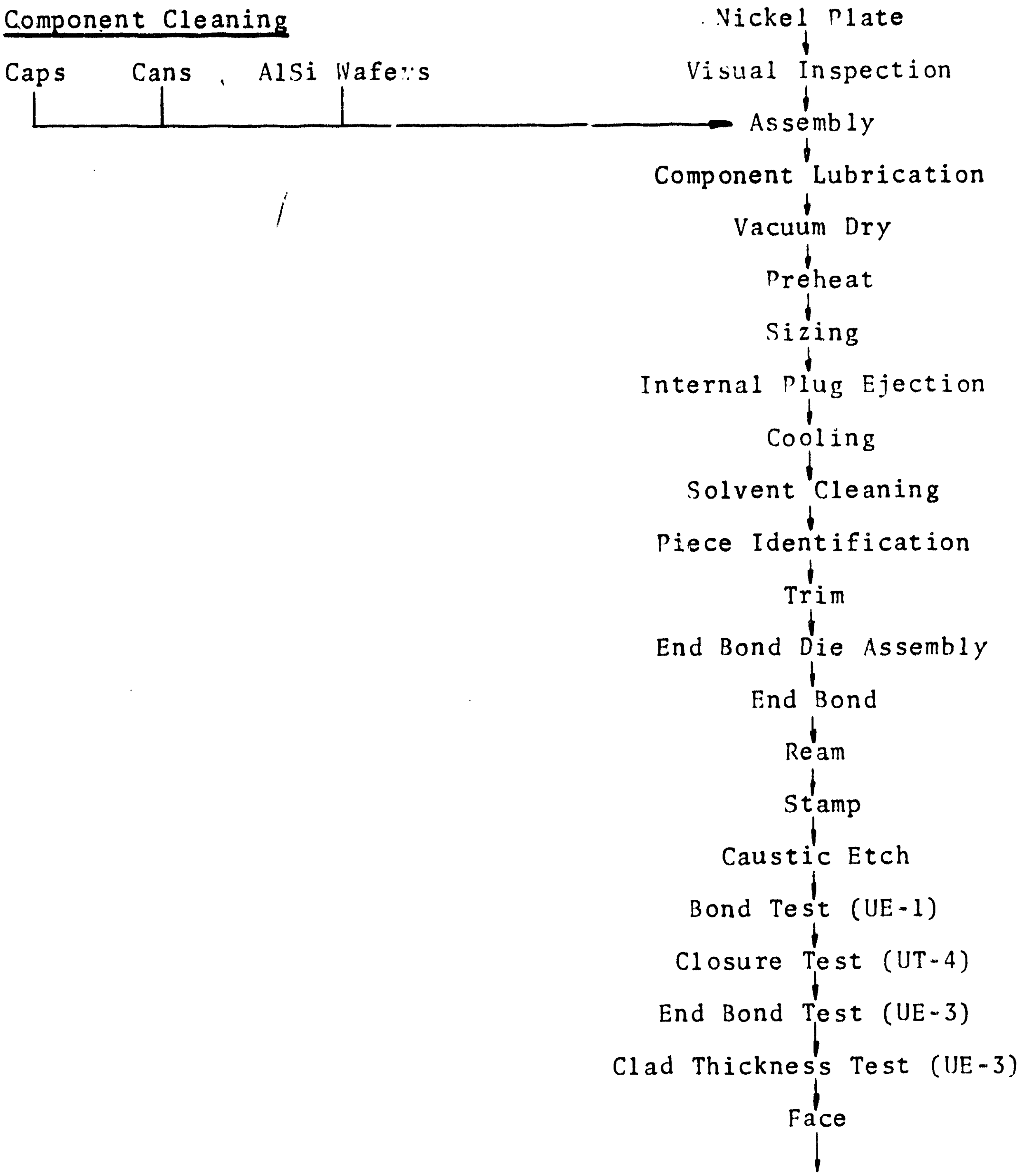

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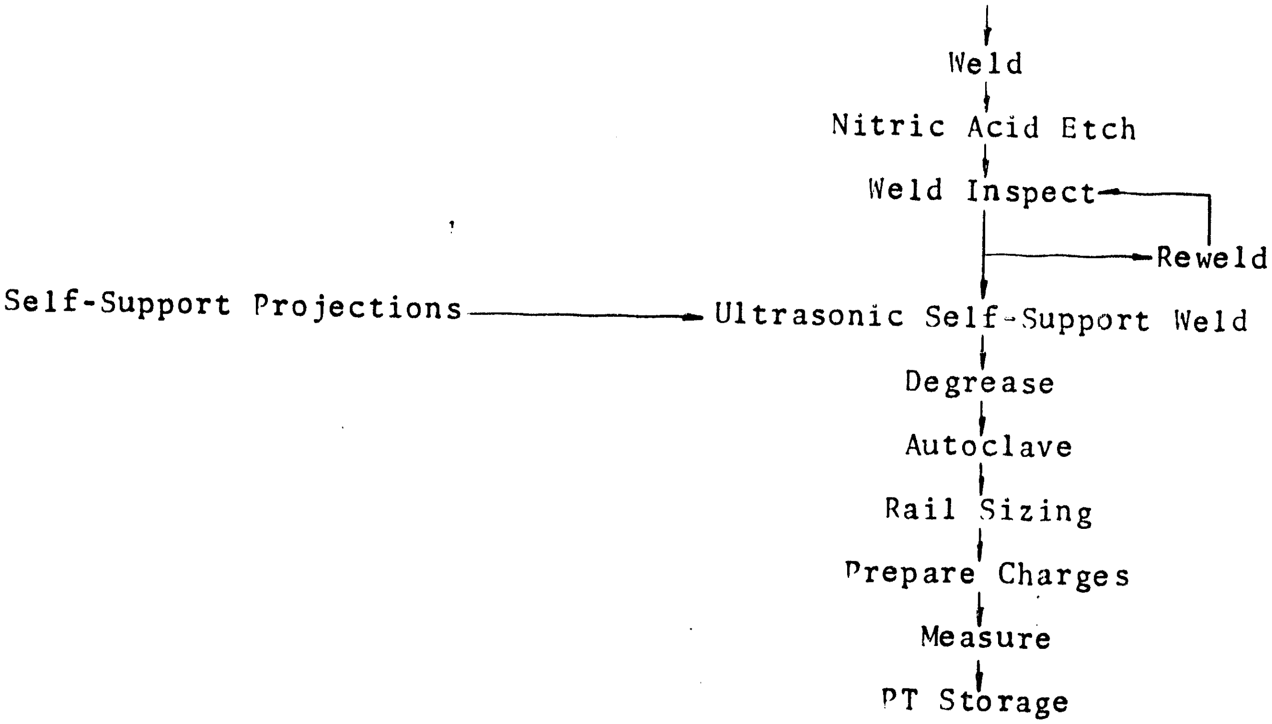
FIGURE 2 (Cont'd)
HDS PROCESS FLOW SHEET FOR OIL AND WATER QUENCHED CORE FABRICATION



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Uranium Core Preparation

Uranium cores for this test consisted of matched water-quenched and oil-quenched cores taken from alternate positions in the core rod stock. The two types of material were each divided in three groups: (1) as received, (2) alpha annealed, and (3) AlSi pretreated. Alpha annealing consisted of holding cores at 550°C in a vacuum of 3-5 x 10^{-5} mm Hg for 1 hour. Cores for the third group were canned by the AlSi brazing process. Because the C-model HDS core is larger than a standard C-size core, the HDS cores were canned in K5N cans and 03N spires. Component cleaning and core preparation consisted only of degreasing. After canning, the assembled pieces were sent directly to core recovery for jacket removal.

Cores for this test had more cracks and striations than normal. For this reason, a large number of cores were rejected at pickle inspection. The crack tester was modified to detect striations and was overly sensitive. From 60 to 100 percent of all the pieces tested were rejected by the UT-2 tester. Because of the high tester sensitivity and the fact that the tester standard has not as yet been firmly established, all pieces rejected by the crack tester were retained for visual inspection prior to nickel plating.

Nickel Plating

Uranium cores were electro-nickel plated with a 0.8 mil nickel plate. Because of the high incidence of striations in the surface of the cores for this test, the inspection for cracks after nickel plating was changed. As yet, no actual standard for inspecting core cracking after plating has been established, but the following limits have been used:

	Piece Reject Limits
Prior to this test:	Any crack over 1 inch long. Any crack with bottom not visible. More than five 1/4 inch cracks.
This test:	Any crack over two inches long. Any crack with bottom not visible.

Component Cleaning

Cap and can components were cleaned by caustic etching and deoxidizing as follows:

1. Degrease - warm trichloroethylene 65-85°C, 1 min.
2. Caustic etch - three to six percent Aluminux plus three to six percent sodium nitrate at 50-60°C. Can outside

[redacted]

diameter reduced 0.005 ± 0.002 in. and cap outside diameter by 0.008 ± 0.002 in. (Aluminux concentration increased to that used for spire etching.)

3. Flowing hot water rinse.
4. Flowing cold water rinse.
5. Dip in caustic etch solution (No. 2 above) 10-20 seconds.
6. Flowing hot water rinse.
7. Flowing cold water rinse.
8. Deoxidize in Diversey 514 (or equivalent) solution, 16 ± 3 oz. per gal. for 10 ± 5 min.
9. Flowing cold water rinse.
10. Second flowing cold water rinse.
11. Methanol rinse.
12. Second methanol rinse.
13. Hot air dry.

A residue resembling hard water scale was noted on some of the can components during the early part of this run. This scale was believed to have been caused by caustic solution drying on the components between the first and second caustic dips (No. 2 to No. 5 above). Some difficulty was experienced in maintaining the low Aluminux concentration of 4 to 5 oz. originally specified. (Corresponds to 2.9 to 3.6 percent.) For this reason, the Aluminux concentration was changed to that used for spire etching.

AlSi wafers prepared from AlSi produced on site were cleaned as follows:

1. Caustic etch - Turco 4181-19, 2 oz. per gal. at room temperature.
2. Flowing cold water rinse.
3. Nitric acid - 20 vol. percent, 2 min. ± 5 sec., cold.
4. Flowing cold water rinse.
5. Nitric-hydrofluoric acid - 30 vol. percent HNO_3, 0.5 vol. percent HF and 69.5 vol. percent water, cold, for 2 to 3 min.

6. Nitric acid - 20 vol. percent HNO_3 for 2 min. ± 5 sec., cold.

7. Flowing cold water rinse.

8. Methanol rinse.

9. Air dry.

With the exception of the upper two inches of a can, all cleaned components were handled with clean cotton gloves. Oil on the outside of a can prevents the graphite lubricant (Aqua Dag) from adhering. The upper three inches were not coated and were used for handling during can-core assembly and lubrication operations.

Preparation for Sizing - Component Assembly, Lubrication, and Vacuum Drying

Nickel-plated cores were assembled in cleaned components immediately after nickel plating. As in previous production tests, an AlSi wafer was used between the cap and core to provide a secondary barrier to water entry on the cap end. Since the AlSi wafer offers no advantage in terms of improved bonding on the ends of the fuel, plans are underway to eliminate the AlSi wafer from the process.

After assembly, the cans were dipped in an Aqua Dag solution (5-6 parts water to 1 part Aqua Dag) and allowed to air dry. The pieces were then placed in the vacuum dryer set at a temperature of 90 to 110°F and held for 30 minutes at a vacuum of 1 mm Hg. At the end of the drying period, the vacuum chamber was back-filled with Argon. Just prior to sizing, the Argon-filled fuel assemblies were fitted in carriers.

Midway in the test, the vacuum pump failed due to a leak in a seal. It was discovered after the vacuum pump oil leaked out overnight. The pump was repaired and the dryer was back in operation in about four hours. Approximately 60 plated cores were in the dryer at the time. These pieces were not lost because of pump failure, but because of the decision to discontinue the run when an apparent bonding problem was indicated.

Sizing

Fuel elements were sized at a rate of one every 2-1/2 minutes for this test. A six-position holder was used in the preheat furnace leaving 5 pieces in the furnace at a time after sizing started. The preheat furnace was set to control temperature at 680°C; however, opening and closing the door caused the indicated temperature to fluctuate 15 degrees above and below the set point.

The sizing reject rate for this test was 9.5 percent. Wrinkled cans amounted to 5.7 percent. Wrinkling occurs when the cap seizes the can during sizing. Wrinkled cans are influenced by press alignment, lower ram height, and core warp, in addition to fuel assembly temperature. When it was determined that a wrinkling problem had developed, the carrier holder was realigned, and the lower ram tip was dropped to permit the piece to position itself in the die before contacting the lower ram. After the above changes were made, wrinkling was reduced from 11.6 percent to 4.1 percent. The 4.1 percent wrinkling reject rate is also considered high and is partly attributed to the core pretreatment which both roughened the surface and warped the cores somewhat. It was originally intended to measure the amount of core warp, but due to the press of time and manpower shortage, it was impossible to do so.

Solvent Cleaning

Stoddard's solvent was used to dissolve the coating of Fiske 604 extrusion lubricant. Removal was improved by using a soaking technique to soften the residual coating followed by brushing the bore and wiping the outside surface. Sized pieces were placed in the solvent bath with the flash down to minimize solvent contact with the cap-can bond area.

After cleaning, core identification number from the cap was marked on the outside of the piece with a wick pen.

Trim

The 3-1/2 inch flash produced on the cap end of hot die sized fuels during sizing was removed by facing. The cap and base ends were also faced to flatten the surface and to permit fitting the end bond die cups and plugs. Approximately .007 in. of material was removed from each end.

During this last run, facing was simplified by providing an 0.010 in. circular indentation on the cap end and an 0.020 in. triangular indentation on the base. The marks also served to identify the ends.

End Bonding

Two induction heated end bond presses powered by a common motor-generator unit were used for this run. They were fitted with Inconel-X heads in place of the stainless steel heads, No. 416, which in previous runs had experienced severe distortion. No distortion of the Inconel-X heads was observed during the current run. The previously used stainless steel heads often required replacement after 50 cycles.

During this run, the end-bonding operation was quite smooth, with a piece being produced every two minutes. The installation of timing equipment permitting fully automatic operation greatly simplified the process and improved uniformity of treatment to each piece.

After fitting each piece with an Inconel-X die cup and an AF-71 die plug, HDS pieces were placed in the end-bonding presses. End bonding was accomplished by applying 4 tons per square inch on the end of the piece for a period of 2 min., 45 sec. Press heads were preheated to 725°C and then heated by induction for a period of 1 min., 30 sec. to bring the heads back to starting temperature. At this time, the power was cut off and transferred to the other press. End bond pressure remained on to complete the end-bonding cycle. End bond process limits for producing material for this production test are listed in the table below.

END-BONDING PROCESS CONDITIONS

End Bond Die Material	
Die cups for outer lateral surface	Inconel-X
Die plugs for inner lateral surface	AF-71
End Bond Press Head Material	Inconel-X
Initial Head Temperature	725°C
Pressure on End of Piece	4 ± 0.25 T/in.^{2}
Total Press Time	2 min., 45 sec.
Induction Unit Power Setting*	Press A-12KW Press B-10KW
Induction Unit Power Time After Pressure Applied at Beginning of Cycle (± 1 sec.)*	Press A - 1 min., 30 sec. Press B - 1 min., 35 sec.

*Although the power settings and times are different, the head temperatures were the same due to the difference in coupling between the individual heads and induction coils.

Immediately after end bonding, a reaming tool was used to remove the slight back extrusion produced close to the fuel ID where the inner die plug and outer die cup fit together. The pieces were then stamped with their individual core numbers.

Cleaning

End bonded fuel elements were cleaned by a nitric acid, caustic, and nitric acid treatment to prepare them for testing and welding. An initial nitric acid treatment was used to remove most of the residual Aqua Dag and Fiske 604 coating followed by a caustic etch to remove 0.0015 in. from the surface. Nitric acid was used to desmut the etched pieces. Solutions and methods used are summarized briefly as follows:

1. Nitric acid - 50-60 w/o HNO_3 at 85-95°C for 20-25 min.
2. Flowing process water rinse.
3. Preheat by immersing for 2-3 min. in 60-95°C water.
4. Caustic etch - Turco 4181-19, 2 to 3 oz./gal., 70-75°C for 4-1/2 to 5 min.
5. Hot flowing water rinse - 50-95°C for 1 min.
6. Nitric acid - 15-30 w/o HNO_3 at 50-90°C for 10-15 sec.
7. Hot flowing water rinse - 50-95°C for 2-5 min.
8. Warm air dry.

Nondestructive Testing

After cleaning, fuel elements for this test were subjected to the following nondestructive tests: internal and external bond tests, cap and base end bond tests, internal and external clad thickness tests, and internal and external closure integrity tests. During previous runs when testing was performed after lubricant removal, traces of lubricant remaining after the solvent cleaning collected on sensing probes making testing difficult. No such problem was encountered in this run since the additional cleaning steps prior to testing removed all surface contaminants.

Early in the run a bonding problem was indicated when the first two trucks of material produced were sent ahead to the bond tester. Sizing was discontinued to evaluate the problem the next day, July 15, when it was discovered that the vacuum pump on the vacuum dryer was broken down. A number of tests were made on the fuel and process equipment. Both metallographic examination and bond strength measurements in the supposed defective areas revealed no unusual conditions. It was concluded that no bonding problems existed, and preparations were made to start processing fuel again.

Nondestructive test data are summarized in Table 1 along with other reject categories. The eight external closure integrity (UT-4) tester rejects were examined and found to be false rejects caused by surface marring. Fourteen pieces were rejected at the cap end bond test and eight at the base end test. Four of each of the cap and base end rejects were found to be false as shown in Table 1. The UE-3 tester was used for clad thickness testing. No clad thickness rejects were detected.

Facing

Hot die sized fuel elements were faced to clean up the ends and provide weld bead relief. The cap end was flat faced and the base end faced as shown in Figure 3.

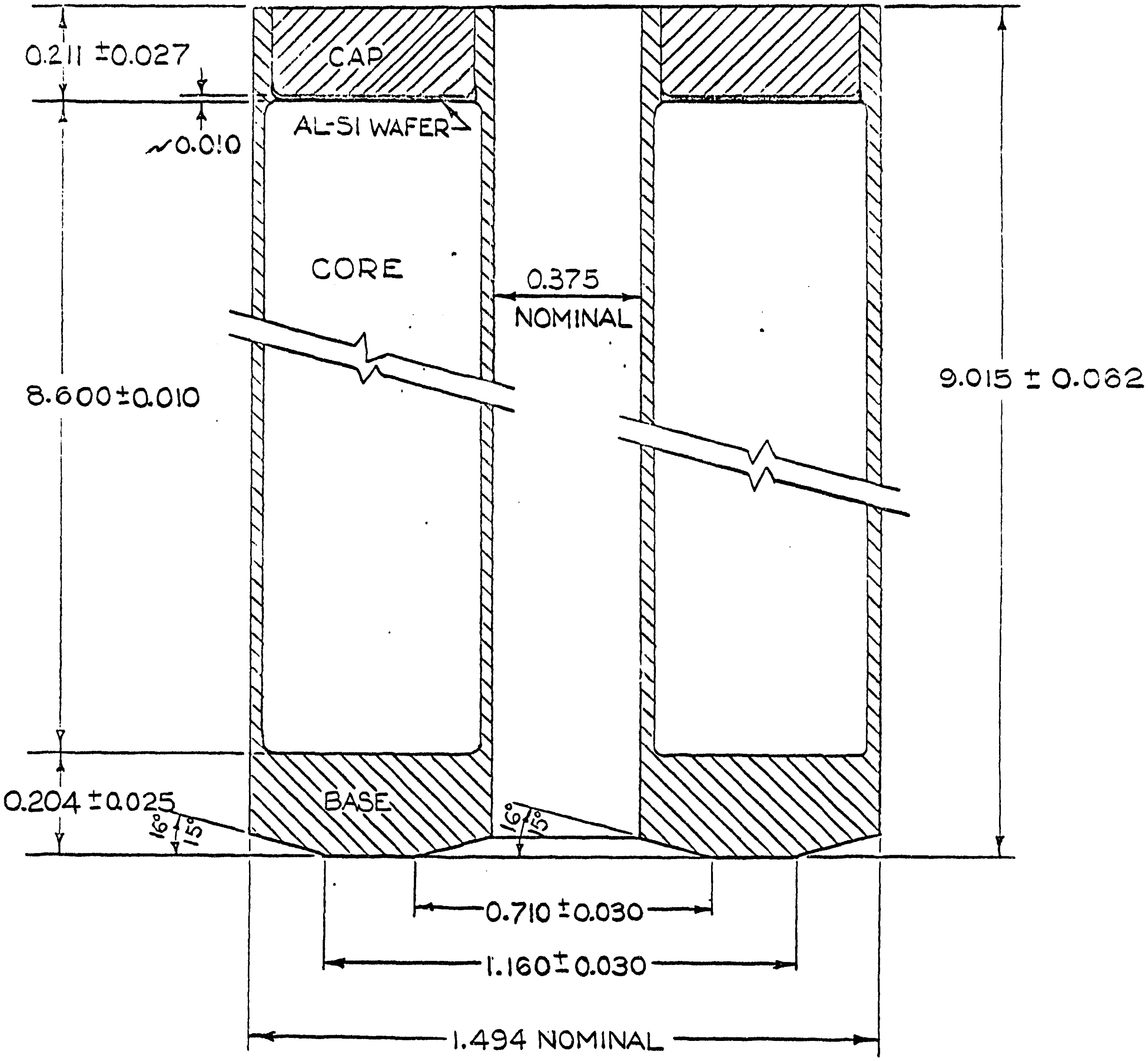

FIGURE 3

Facing Instructions for Model CDB2N Fuel Elements

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Welding and Weld Inspection

Welding of the hot die sized fuel was routine, using two presses with an inert gas mixture of 95 percent helium and 5 percent argon at a turntable speed of 20 rpm. Welder amperages for the external and internal welds were 140 and 125 amps minimum, respectively.

Prior to inspection, the fuel elements were etched to remove oxides which could be covering potential defects. Etching consisted of a 20-30 second dip in Turco 4181-19 at 70-75°C followed by a hot process water rinse and a 10-15 minute nitric acid (15-30 w/o) etch at 80-90°C. The pieces were then rinsed and dried.

No unusual problems were encountered with welding. Any piece having a small but questionable defect was rewelded and reinspected.

Self-Support Projection Attachment

Self-support projections were ultrasonically welded to the HDS fuel elements according to the standard procedure for attaching supports to Model C5N AlSi brazed fuel. Because the finished fuel is slightly longer, the self-support projection rows were moved down from the cap 1/8 inch. Projections were attached 1-3/8 ± 1/16 inch and 5-3/8 ± 1/16 inch from the ends. After ultrasonic welding, the finished pieces were degreased to remove oil used to aid welding.

Autoclave

Hot die sized fuel elements were autoclaved for a minimum of 50 hours. The pieces were actually in autoclave over a weekend. A long autoclave time has been used to assure fuel jacket and closure integrity by permitting gross rupturing in the event of water entry to the uranium core.

Rail Sizing

Rail heights were reduced by 0.005 in. to compensate for growth during irradiation. Minimum rail height was 0.077 in. and the maximum circumscribed circle was 1.656 in.

Preparation of Test Charges

A total of 18 charges was prepared from hot die sized fuel produced for this test. Charge make-up is shown in the test design supplied by K. B. Stewart.* (See Appendix.) Every effort was made to keep pieces in the prescribed order, however, due to the use of six variables from single ingots, it was difficult to do so in all cases. In the event a piece was unavailable, another piece from a large test group from the same ingot was used. Whenever a substitution was made, it was recorded on the pre-irradiation data sheets.

Pre-irradiation measurement data have been submitted for statistical analysis and will be reported as a supplement to this report.

Metallography

Twelve fuel elements, two from each test condition, are being prepared for metallographic examination. Due to the time required for sectioning and evaluation, results of metallographic examination will be reported as a supplement to this report.

C. A. Strand

Engineer
Process Development Unit
Fuels Engineering Operation
Production Fuels Section, IPD

CA Strand:nbh

*K. B. Stewart, Letter to C. A. Strand, "A PT Design for a 2x3 Factorial Test," dated June 18, 1964.

HANFORD LABORATORIES
GENERAL ELECTRIC
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99352

ON

APPENDIX

June 18, 1964

C. A. Strand
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A PT DESIGN FOR A 2X3 FACTORIAL TEST

Fuel elements, differing in the following characteristics of manufacture, are used in this test:

Treatment	Method of Fabrication
A	Oil Quenched - Regular Treatment
B	Oil Quenched - α Annealed
C	Oil Quenched pre AlSi dip treatment
D	Water Quenched - Regular Treatment
E	Water Quenched - α Annealed
F	Water Quenched pre AlSi dip treatment

or

	Regular Treatment	α Annealed	Pre AlSi Dip Treatment
Oil Quenched	A		C
Water Quenched	D		F

The arrangements of fuel elements within units are as follows:

Position	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6
5	A	E	C	B	D	F
6	B	C	E	A	F	D
7	C	D	A	F	E	B
8	D	F	B	C	A	E
9	E	B	F	D	C	A
10	F	A	D	E	B	C
11	B	F	D	A	E	C
12	A	D	F	B	C	E
13	F	B	E	C	D	A
14	C	E	A	D	F	B
15	D	A	C	E	B	F
16	E	C	B	F	A	D
17	D	F	A	C	B	E
18	F	E	B	A	C	D
19	B	D	F	E	A	C
20	A	C	D	B	E	F
21	E	B	C	D	F	A
22	C	A	E	F	D	B
23	B	E	C	D	A	F
24	C	D	A	F	B	E
25	A	C	E	B	F	D
26	E	F	B	A	D	C
27	F	A	D	E	C	B
28	D	B	F	C	E	A

The purpose of a designed production test is to place the fuel elements in the tubes according to a predetermined pattern in a way which enables us to make comparisons between treatments which are unbiased and which enables us to assess whether these differences are statistically significant by valid procedures. Here there are 6 different treatments representing a 2X3 factorial design. Random assignment of fuel elements would result in unbiased comparisons. However, this would be very inefficient. The large amount of variation in incremental dimensional distortion values due to the flux effect would be part of the residual error. Any pattern which eliminates this effect --- or allows us to subtract it out will result in a much smaller residual error. The following design has this property. Assume that the flux effect on Δ dimensional values is linear. (A tenable assumption even if not exactly correct). Assign flux effects of 1,2,3,..., 12, 12, 11, 10, ..., 3, 2, 1 for positions 5, 6, 7, ..., 28. Then note that in tubes 1 & 2 (tube 2 - positions 5-16) we have

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ACCENT ON VALUE

Flux Effect

Treatment	A	1 + 8 + 9 + 4 + 6 + 11 = 39
	B	2 + 7 +10 + 6 + 5 + 9 = 39
	C	3 +10 + 7 + 5 + 2 + 12 = 39
	D	4 +11 +12 + 1 + 3 + 8 = 39
	E	5 +12 + 8 + 3 + 1 + 10 = 39
	F	6 + 9 +11 + 2 + 4 + 7 = 39

Thus in comparing the treatment averages they are all subjected to comparatively the same average flux conditions if the tubes are at approximately the same power and exposure. Even the fact that the flux (more exactlyits effect) is not linear is obviated this design if banks of 6 tubes are closed which are similar in regards to power and residence time effects. For this reason it is recommended that at least two such banks and at most 4 such banks are chosen for the experiment. I would tend to conservatism in the sense that 4 banks is not unduly expensive if no treatment is disastrously bad, and if the added work does not increase the chance of some gross error of carrying out the experiment.

KB Stewart

Statistics Operation
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