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END CLOSURE OF
HOT DIE SIZE DIFFUSION
BONDED FUEL ELEMENTS

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END CLOSURE OF
HOT DIE SIZE DIFFUSION
BONDED FUEL ELEMENTS

January 14, 1964

by:

C. A. Strand
 Process Development Unit
 Fuels Engineering Operation
 Production Fuels Section, IPD

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INTRODUCTION

Studies initiated at Hanford in 1961 for the purpose of developing an alternate cladding process for I&E fuel elements for the eight existing production reactors indicated that the hot die sizing diffusion bonding process offered the greatest incentive. Hot die sizing was the most attractive with respect to improved fuel quality and potential reduction in fuel element unit cost when compared to the existing AlSi brazing process.

Initial development work consisted of determining optimum process parameters for producing good diffusion bonds on the inner and outer lateral surfaces during sizing. This report summarizes the results of a series of end bonding variables tests designed for producing good diffusion bonds on the ends of hot die sized fuel elements.

SUMMARY

A process for end bonding hot die sized fuel elements and insuring a high quality closure has been developed. End bonding parameters were developed by running a series of statistically designed tests. A 0.010 inch AlSi wafer (10 per cent silicon-aluminum alloy) was later inserted between the end caps and core to improve bonding between the cap and core. Recommended process conditions for end bonding hot die sized fuel elements using the differential thermal expansion die method for both eight-inch and six-inch fuel models are listed as follows:

	<u>Eight-Inch</u>	<u>Six-Inch</u>
Supplemental Preheat Temperature Setting	680 C	680 C
Assembly Preheat Time $\pm$ 15 Seconds	5-3/4 Min.	4-3/4 Min.
Press Head Temperature $\pm$ 5 C	655 C	655 C
Pressure $\pm$ 0.25 T/in. ²	4 T/in. ²	4 T/in. ²

Initial end bonding development work indicated that the first end bonding press using the interference fit containment die concept left much to be desired in fuel quality and appearance. Results of the first variables test designed to test the effect of time, temperature, and pressure upon end bonding revealed that increased temperatures and pressures as well as a press modification would be necessary to produce good end bonded fuel. Considerable clad upsetting and end bond deterioration was observed on the lateral surface adjacent to the containment die.

The press was re-designed to use the differential thermal expansion die concept for applying pressure to the inner and outer lateral surfaces near the fuel ends. A second variables test designed on the basis of results from initial tests produced much improved end bonding. The tentative process conditions were selected for the initial reactor test material as follows:

Preheat Time in Furnace Set at 680 C	5 Minutes $\pm$ 10 Seconds
End Bond Press Head Temperature	640 $\pm$ 10 C
Pressure	4 $\pm$ 0.25 T/in. ²
Bonding Time	4 Minutes $\pm$ 10 Seconds

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The first production test material end bonded according to the above limits experienced three failures in autoclaves. Consequently, development work shifted to improving the closure zone. Welding technique was modified to give more cover over the cap-can junction and an AlSi wafer was inserted between the core and end caps. The AlSi wafer was to serve as a bonding medium and a secondary barrier in the event of water penetration through the closure. Process parameters were changed to insure further improvement in end bonding at the cost of a small reduction in lateral surface bond strength.

DISCUSSION

Fabrication of fuel elements by the hot die sizing (HDS) process consists of sizing a preheated fuel assembly to bond the core and cladding while producing the desired dimensions. Lateral surface bonding is accomplished by passing the heated fuel assembly through a cold die while passing a die plug through the bore to form the internal bond. End bonding is produced in a later step by applying heat and pressure to the ends of hot die sized fuel elements.

Hot die sized fuel element fabrication starts by assembling nickel plated uranium cores in cleaned components. The external surface is then coated with Aqua-Dag for lubrication during sizing. The lubricated fuel assemblies are dried for 30 minutes at a temperature of 100-125 F at a vacuum of 1 mm Hg and are then filled with argon by backfilling the vacuum chamber. Each piece is then fitted in a carrier, commonly called a "heat shield" and is ready for sizing. Fuel elements for end bond test work were sized according to conditions determined by a sizing test previously reported.⁽¹⁾ Sizing conditions were changed when equipment modification became necessary. Since this report summarizes several end bonding tests, process conditions as well as other pertinent information will be listed under the appropriate headings within each test discussed.

Diffusion bonding by the HDS process occurs in two steps. Nickel-uranium diffusion bonding takes place over the entire piece in the furnace when the assembly is preheated for sizing. With the exception of 1/4 to 1/2 in. from each end, aluminum-nickel bonding on the inner and outer lateral surfaces is accomplished at the time of sizing. Aluminum-nickel diffusion continues until the interface temperature drops below 350 C. The amount of nonbonding on each end is affected by aluminum and nickel oxide formation and chill rates from the dies and ram tips.

The end bond press was designed to accomplish both end bonding and lateral surface bonding near the ends. Lateral surface bonding near the ends was achieved by causing an interference fit between the fuel piece and the die. Test work with the original end bond press utilizing a large containment die left something to be desired with respect to fuel quality, control, and appearance. After the first test, the process was modified to use a differential thermal expansion die concept for inner and outer lateral surface bonding while applying end pressure. Two basic end bonding concepts were tested and the methods used were somewhat evolutionary in nature. For this reason, the tests will be discussed in chronological order under separate headings. Schematic

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diagrams of the original end bond press and the differential thermal expansion die concept are shown in Figures 1 and 2, respectively.

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FIGURE 1

Schematic Diagram of Containment Die End Bonding Press

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FIGURE 2

Schematic Diagram of Differential Thermal Expansion Die End Bonding Press

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END BONDING TESTS 1 AND 2

End bonding tests 1 and 2 were run concurrently. Bonding temperature was obtained by two methods: (1) by heated heads on the end bond press and (2) by preheating the fuel in the resistance furnace prior to pressing in the heated end bond press. Cap-core interface temperatures of 500 C were reached in about eight minutes. Using this long a press time would result in a very low throughput for the end bonding press. For this reason, auxiliary preheating by placing the pieces in the HDS air resistance furnace was tested. Depending upon final desired temperature and preheat conditions, equivalent cap-core interface temperatures could be obtained in about two minutes of press time, which would considerably increase throughput. A comparison of end bond cap-core interface temperatures for pieces heated by the end bond press and by use of a five minute preheat is shown in Figure 3.(2)

Due to a shortage of cores and a desire to test for the effect of differences in uranium, both ingot and dingot cores were used for the end bonding test. The cores were nickel plated in a Watt's plating bath, which is described in HW-77759, pages 122-130.

Hot die sized fuel elements for the end bonding test were sized using the following conditions based upon results of the hot die sizing variables test:(1)

Preheat Furnace Temperature	680 $\pm$ 10 C
Preheat Furnace Time	10.0 $\pm$ 0.5 min.
Wall Reduction External	36 per cent*
Ram Speed	70 $\pm$ 5 in./min.

*Optimum wall reduction 55 per cent, but above used because thick wall components unavailable.

A 36 per cent wall reduction was obtained by using No. 2 Ferrotic C internal dies and the No. 6 Ferrotic C external dies described in Drawing Numbers H-3-17424 and H-3-17425, respectively. The external Ferrotic C die was discovered to be broken towards the end of the sizing run and the last 20 per cent of the pieces prepared were sized with the No. 6 tool steel die.

After sizing, the pieces were faced on both ends. The four inches of flash produced at the cap during sizing was trimmed, and the base was faced to allow the end bond press to accept the piece.

Variables studied in the two parts of this end bonding test are listed below:

Test Variables

	Test 1 Preheat 680 C for 5 Min.	
Press Head Temperature	560, 600, and 640 C	560, 600, and 640 C
Press Time	30, 105, and 180 sec.	5, 7, and 9 min.
Pressure	1.03, 1.38, and 1.72 T/in. ²	1.03, 1.38, and 1.72 T/in. ²

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FIGURE 3

Cap-Core Interface Temperatures for HDS Fuel Elements During End Bonding with and without Auxiliary Preheat

Two pieces were run at each of the prescribed test conditions in the sequence detailed in Appendix A. Each test was repeated once.

Sized pieces for Test 1 above were placed in the resistance furnace for five minutes, transferred to the end bond press, and held for the prescribed time. Pieces for Test 2 were placed directly in the end bond press. After end bonding, the pieces were air cooled to room temperature.

End bonded pieces had considerable galling under the die and a ring of upset metal just outside the containment die. This upset metal was influenced by press alignment and buildup within the containment die. Effects upon fuel quality will be discussed in the bond strength and metallography sections.

End bonded pieces were covered with a residue of Aqua-Dag and Fiske 604 lubricants which was very adherent and difficult to remove. Organic solvents tried heretofore were not capable of cutting the film without considerable hand scrubbing. A solution of nitric and hydrofluoric acids was used to clean off the residue to permit better handling.

Testing and Evaluation of Results

Evaluation of test pieces consisted of destructive and nondestructive testing. Nondestructive tests included internal and external bond testing and cap and base end bond testing. Destructive tests included bond strength measurements by stud pulling and metallographic examination of sections taken from test pieces.

Ultrasonic Bond Count

None of the process variables significantly influenced the total internal bond count for either the preheated or non-preheated test as was the case for the external bond count for the non-preheated test.⁽⁴⁾ An interaction between press time and temperature existed for external bond count for the preheated test. Pressure did not influence the total bond count. Total bond count for the high temperature level increased slightly as time increased, but bond count decreased for both the low and intermediate temperature levels.⁽⁵⁾ The effects of test variables upon external total bond count are illustrated in the predicted response surface shown in Figure 4.

Cap and Base End Bond Count

All process variables influenced the cap and base end bond counts for both the preheated and non-preheated tests. The pieces were tested on an ultrasonic device similar to the existing ultrasonic bond testers except that it was equipped with test heads designed to locate cracks between the cap and base end cladding and the core. In general, end bonding is improved by using higher temperatures, longer press times, and higher pressures. It will be noted that there are some deviations to the previous statement for the base ends of pieces in both tests in the predicted response surfaces shown in Figures 5, 6, 7, and 8. These deviations were due to the variation of oxide formation on the base end of cores and cans. Combinations of higher pressures and longer times helped to improve lower temperature material by overcoming oxide formation.

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FIGURE 4

Predicted Response Surface of Total External Bond Count for the Preheated End Bond Test Showing the Effects of Time, Temperature, and Pressure

FIGURE 5

Predicted Response Surface of Total Cap End Bond Count for the Preheated End Bond Test Showing the Effects of Time, Temperature and Pressure

FIGURE 6

Predicted Response Surface of Total Base End Bond Count for the Pre-heated End Bond Test Showing the Effects of Time, Temperature, and Pressure.

FIGURE 7

Predicted Response Surfaces of Total Cap End Bond Count for the Non Preheated End Bond Test Showing the Effects of Time, Temperature and Pressure.

FIGURE 8

Predicted Response Surface of Total Base End Bond Count for the Non Preheated Test Showing the Effects of Time, Temperature and Pressure.

External Bond Strength

External bond strength measurements were made on test fuel elements by welding ten 1/4 inch diameter studs to each of the pieces. Two rows of five studs spaced as listed below were pulled from each piece 180 degrees apart. Average bond strength measurements for the preheated, non-preheated, and control (sized but not end bonded) pieces are listed below:

Average Bond Strength Measurements Expressed as Pounds Force to Pull a 0.25 In. Diameter Stud

<u>Position</u>	<u>Location</u>	<u>Preheated Test</u>	<u>Without Preheat Test</u>	<u>Control (Not End Bonded)</u>
1	3/4 in. from cap	740	778	296
2	1-1/2 in. from cap	585	460	862
3	Center	931	901	907
4	1-1/2 in. from base	570	450	874
5	3/4 in. from base	935	909	531

Note that end bonding increased bond strength at the positions 1 and 5, which were 3/4 inch from the ends while reducing it adjacent to the die. Bond strength at the center was not appreciably changed.

Average bond strength at positions 1, 3, and 5 are high and were not affected by the test conditions. Bond strength at positions 2 and 4 were lower and affected by time and temperature. In general, lower levels of time and temperature produced the highest bond strength.(6) Predicted response surfaces of bond strength for positions 2 and 4 for the preheated and non-preheated test are shown in Figures 9, 10, 11, and 12. In the case of position 4 for the preheated test, pressure also influenced bond strength. Highest bond strength was produced at the low pressure, low temperature and low time levels as shown in Figure 10.

No significant difference between ingot and dingot uranium cores was detected.(7) Average bond strength measurements taken at the five previously mentioned positions are listed below for the ingot-dingot test of Replicate 3.

Bond Strength Measurements for the Ingot-Dingot Comparison-- Expressed as Pounds Force to Pull a 0.25 in. Diameter Stud

<u>Position</u>	<u>Location</u>	<u>Ingot Average</u>	<u>Dingot Average</u>
1	3/4 in. from cap	893	868
2	1-1/2 in. from cap	795	768
3	Center	846	798
4	1-1/2 in. from base	716	753
5	3/4 in. from base	1014	997

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FIGURE 9

Predicted Response Surfaces Showing the Effects of Test Variables upon Bond Strength Adjacent to the Containment Die, 1-1/2 in. from Cap for the Preheated Test.

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Predicted Response Surfaces Showing the Effects of Test Variables upon Bond Strength Adjacent to the Containment Die, 1-1/2 in. from the Base End for the Preheated Test.

FIGURE 10

FIGURE 11

Predicted Response Surfaces Showing the Effect of Test Variables upon Bond Strength Adjacent to the Containment Die, 1-1/2 in. from the Cap End, for the Non Preheated test.

FIGURE 12

Predicted Response Surfaces Showing the Effects of Process Variables upon Bond Strength Adjacent to the Containment Die, 1-1/2 in. from the Base End, for the Non Preheated Tests.

Metallography

Representative samples from each of the test conditions were submitted to the Analytical Laboratory for sectioning and polishing. A diagram of locations where samples were taken is shown in Figure 13. Nickel-aluminum diffusion layers produced resembled a typical hot press bond, especially at the high time and temperature levels. Diffusion thickness, both Al-Ni and Ni-U, increase as time and temperatures increase. Typical diffusion bonds are shown in Figure 14. The lower photomicrograph shows how an oxide layer prevents Al-Ni diffusion.

Best end bonding was produced on the base end because of less nickel oxide present. Nickel oxide formation at the cap and end was more pronounced because chilling caused air to be drawn into the fuel assembly prior to and during sizing. Continuity of end bond diffusion layers depended upon the degree of contact between the jacket end cap and core as limited by oxide or achieved by pressure. The most continuous bonds were produced by high temperatures and high pressures in this test.

The severity of cracking within the Al-Ni diffusion layer at the previously mentioned ring of upset metal just outside of the containment die increased at high levels of time and temperature. As had been previously mentioned, cracking influenced the bond strength. A typical cracked Al-Ni diffusion layer is shown in Figure 15. In order to evaluate more fully the extent of cracking, a simple grading system was set up whereby the amount of cracking was estimated. Response surfaces showing the extent of internal bond cracking due to process variables are shown in Figure 16 for the preheated test. Although the response surface in Figure 16 may appear odd, it agrees quite well with observations made of the sectioned pieces. There was a noticeable improvement in bonding for the low temperature level (560 C) as time increased. The poor bonding at the short time was actually non-bonding. Time did not influence bond cracking at the intermediate level--600 C. At the high temperature, 640 C, best bonds were produced at the short time. At longer times, the diffusion layers became thick and more susceptible to fracture as stress was applied. Variation in pressure did not influence cracking in this case.

Estimating the amount of cracking was a problem in that it was difficult to determine whether or not a flaw was a crack in a thin bond or the lack of a bond. No differentiation was made between the two because both were detrimental to the bond, which accounts for the peculiar response surfaces.

Aluminum-nickel diffusion cracking for the internal bond in the non-preheated test is shown in the predicted response surface of Figure 17. In general, less cracking can be expected at the lower temperature level. Interactions between time, temperature and pressure are more complex and are undoubtedly influenced by oxide formation as in the preheated case. Note that the time scale was reversed to simplify drawing the response surfaces.

A similar technique of measuring cracks within the cap and can bond was used to grade closure integrity. None of the process variables significantly influenced closure integrity for the inner and outer bond of the preheated test.

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Longitudinal Sections Along Diameter of A, A-2, C and C-2
Polished. Transverse Pie Section From B Polished.

FIGURE 13

Instructions for Sampling End Bonded HDS Fuel Elements for Metallographic Examination

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7026A

1000X

7024A

1000X

FIGURE 14

Typical Diffusion Bonds on End Bonded HDS Fuel for Test 1 and 2. Lower Photomicrograph Shows the Effect of an Oxide Layer Preventing Al-Ni Diffusion.

Cracked Diffusion Layer

6910-C-2

1000X

FIGURE 15

Typical Cracked Al-Ni Diffusion Bond

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FIGURE 16

Predicted Response Surfaces Showing the Effect of Test Variables upon Al-Ni Diffusion Cracking on the Internal Surface of Preheated End Bonded HDS Fuel Elements.

FIGURE 17

Predicted Response Surfaces Showing the Effects of Test Variables upon
Al-Ni Diffusion Cracking on the Internal Surface of Non Preheated
End Bonded HDS Fuel Elements

or for the inner bond of the non-preheated test.⁽⁸⁾ However, all process variables affected the outer closure zone on the non-preheated test. As will be noted in Figure 18 showing predicted response surfaces for the various temperatures, the surfaces are quite complex and there appear to be some inconsistencies. In general, it may be stated that higher temperatures at higher pressures and longer times produced the best bond between cap and can.

Conclusions

Based upon results of Tests 1 and 2, it was concluded that end bond pressure would have to be raised to increase the amount of contact between the jacket ends and the core. Upsetting just outside the containment die would have to be eliminated from both an appearance and bond integrity standpoint. Results of this test showed that both higher temperatures and higher pressures as well as methods for producing a better appearing fuel element would be necessary.

As soon as preliminary results were available, equipment design changes to alleviate the upsetting and Al-Ni cracking were initiated. Plans were made to test the use of differential thermal expansion dies to bond the lateral surface near the ends while using the press to bond the ends. Results of test work using this concept are discussed in the following section.

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FIGURE 18

Predicted Response Surfaces Showing the Effects of Test Variables upon External Cap-Can Closure Integrity for the Non Preheated End Bond Test

END BONDING TEST NO. 3
(Differential Thermal Expansion Dies)

End bonding test No. 3 differed from the first two tests in that lateral surface bonding near the ends was accomplished by pressure produced by the difference in thermal expansion between the end dies and the fuel element. End bonding on the flat end surface was obtained in the same manner as in the previous test. Fuel elements were fitted with die cups and die plugs while cold. Die cups were machined from Inconel-X and die plugs were made of Alleghany Ludlum AF-71 (See Figure 19). The cups had a lower coefficient of expansion while the die plugs had a higher expansion than the hot die sized aluminum-clad uranium fuel core. Upon heating, the difference in expansion produced pressure on the lateral surface near the ends. A diagram of end bonding by the differential thermal expansion concept was shown in Figure 2.

Due to the increased mass caused by adding die cups and plugs to the piece, it was necessary to measure the core and end cap interface temperature. A temperature curve for a piece fitted with end bond dies preheated in the resistance furnace set at 680 C for 5-1/2 minutes and end bonded at 600 psig (5.18 T/in.²) with a head temperature of 620 C is shown in Figure 20.⁽⁹⁾ Note that the rate of heating was slower than that shown in Figure 3 for pieces without die cups, but that the temperature did not drop during piece transfer from the resistance furnace to the end bond press.

Hot die sized fuel elements for the differential thermal expansion end bonding test were sized and handled up to end bonding according to the same conditions used for the initial end bonding test except that the preheat time was changed to 12.0 ± 0.5 minutes. Removal of the circulating fan, which had been giving considerable mechanical trouble, made it necessary to increase the preheat time by two minutes. Additional time was required to compensate for a lack of circulation which reduced the heating rate. Pieces for this test were sized through the No. 5 external die shown in Drawing No. H-3-17425 to give a 45 per cent wall reduction. Internal sizing was the same as in the first test. Core nickel plating is described in detail in HW-76992.⁽¹⁰⁾

Variables studied for the differential thermal expansion test were:

Press Head Temperature	610, 640, and 670 C
Press Time	2.5, 3.5, and 4.5 min.
Pressure	3, 4, and 5 T/in. ²

Three pieces were run at each set of test conditions. The test sequence⁽¹¹⁾ is described in detail in Appendix B.

Sized pieces fitted with end bonding die cups and plugs were placed in the resistance furnace for five minutes, loaded in the end bonding press, and end bonded at the specified conditions. After bonding, the pieces were water quenched and the die cups and plugs removed. The fuel core contracted sufficiently to permit removal of the end bond dies.

End bonded pieces for this test did not have the large amount of upset metal characteristic of the first test. Just prior to running the test, a slight amount of upsetting was observed, but was eliminated by aligning the press heads.

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Inconel-X Die Cup

AF-71 (Alleghaney Ludlum) Die Plug

FIGURE 19

Differential Thermal Expansion End Bond Dies

FIGURE 20

Cap-Core Interface Temperature During End Bonding HDS Fuel
Elements Fitted with Differential Thermal Expansion Dies

Testing and Evaluation of Results

Evaluation of this test was quite similar to that of the previous tests except that additional tests were added. Nondestructive testing included internal and external bond testing, cap and base end bond testing, and dimensions. Destructive testing included inner and outer bond strength measurement by stud pulling and metallographic examination of diffusion bonding.

Ultrasonic Bond Count--Inner, Outer, Cap and Base

Fuel elements were tested on prototypic ultrasonic bond test equipment in a manner similar to that of the previous tests. None of the process variables had any statistically significant main effects upon internal and external total bond counts or cap and base end total bond counts.(12)

Perhaps of most significance was the fact that there was no effect upon cap and base end bonding. After the initial test, data were extrapolated and it had been predicted that a minimum of 300 psig (2.58 T/in.²) would be required to assure intimate contact between cap and core while end bonding.

Dimensions

Fuel elements from this test were measured according to the procedure used for measuring fuel elements for reactor testing. Inner and outer dimensions were taken at three locations listed below:

	<u>Dimension</u>	
	<u>Inner</u>	<u>Outer</u>
1 in. from base	ID-1	OD-1
Center	ID-2	OD-2
1 in. from cap	ID-3	OD-3

Warp measurements taken at the center of the piece are complicated by variations in ellipticity. Statistical analysis of the data(13) indicate that OD-1, OD-2, and OD-3 maximums increase with higher pressures. Minimum diameters at OD-2 decrease with increased time, consequently, ellipticity, expressed as OD maximum minus OD minimum, increased with both time and pressure. Ellipticity increased with time for OD-3. Warp occurs with longer times and higher pressures. Predicted effects of test variables upon external dimensions are shown in Figure 21. None of the test variables influenced internal dimensions.

External Bond Strength

External bond strength measurements were made by the stud pull method similar to that of the first end bond tests, except that smaller studs, 0.190 in diameter, were used. Statistical analysis(14) of bond strength data shows that higher temperatures and longer times give higher bond strength at points 3/4 in. from each end. The opposite is true for locations outside the die cups, 1-1/2 in. from each end. There were no main effects at the center of the piece. Predicted response surfaces for each of the five locations where bond strength measurements were taken are shown in Figure 22.

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FIGURE 21

Predicted Effects of Test Variables, Pressure and Time, upon Finished Fuel Maximum Diameter, Ellipticity and Warp for the Differential Thermal Expansion End Bond Test.

[REDACTED]

POSITION NO. 1
3/4 in. From Cap

PC
1-1

POSITION NO. 2
1/2 in. From Cap

POSITION NO. 3
Center

POSITION NO. 4
1-1/2 in. From

FIGURE 22

Predicted Response Surfaces for External Bond Strength Showing the Effect of Test Variables at Five Locations on an End Bonded HDS Fuel Element Using Differential Thermal Expansion Dies.

POSITION NO. 4
1-1/2 in. From Cap

POSITION NO. 5
3/4 in. From Base

Showing the
d HDS Fuel
s.

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Internal Bond Strength

Internal bond strength measurements were taken by welding 0.125 in. diameter studs to the inner surface of HDS fuel elements cut in two. Except at one location, 1-1/2 in. from the cap, none of the process variables affected internal bond strength.⁽¹⁵⁾ The pressure and pressure-temperature interaction for this location was considered spurious. Average internal bond strengths for the internal bond of test pieces is listed below.

Internal Bond Strength Measurements Expressed as Pounds Force to Pull a 0.125 in. Diameter Stud

<u>Position</u>	<u>Location</u>	<u>Pounds Force</u>
1	3/4 in. from Cap	247
2	1-1/2 in. from Cap	238
3	Center	280
4	1-1/2 in. from Base	259
5	3/4 in. from Base	303
<u>Piece Av.</u>		<u>265</u>

Metallography

Samples taken from each of the test runs were prepared by the Analytical Laboratory for metallographic examination. Each section, as shown in Figure 23, was examined for continuity and thickness of the Al-Ni diffusion bond. To aid in evaluation of the effects of process variables upon end bonding, each sample was examined at points shown in Figure 24 for Al-Ni diffusion thickness and graded.

Statistical analysis of data taken for Al-Ni diffusion thickness showed that diffusion thickness generally increased as time and temperature increased while diffusion was not consistently affected by pressure.⁽¹⁶⁾ Time had a more consistent and pronounced affect than temperature on diffusion thickness. Response surfaces showing the affect of process variables upon Al-Ni diffusion thickness measurements taken at points 3, 7, and 9 (Figure 24) for the base and cap ends are shown in Figures 25 and 26, respectively. Variation noted in the response surfaces was believed to have been caused by oxide formation, oxide penetration and subsequent Al-Ni diffusion by a complex interaction between the process variables.

Predicted response surfaces of diffusion thickness at points about 5/8 inch and 1-1/2 inch from the base and cap ends (Points ID₁₀ and ID₁₈, respectively, for Sections "B" and "D" of Figure 24) are shown in Figures 27 and 28. Diffusion thickness increased with time and temperature and was unaffected by pressure. At the point ID₁₀, closest to the end cap, Al-Ni diffusion was thicker because it was closer to the heated press head. Point ID₁₈ was about 3/8 inch below the die plug.

Response surfaces for Al-Ni diffusion thickness of the outside bond are shown in Figures 29 through 32. Due to the complexity of the response surfaces

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- Sections "A" and "E" - Longitudinal Section Through Center
- Sections "B" and "D" - One Side of Longitudinal Section Through Center
- Section "C" - Save for Additional Sectioning

FIGURE 23

Instruction for Sectioning HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies.

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*Al-Ni Diffusion Layer Thickness Measurements Taken. (Maximum Observed in Field).

FIGURE 24

Location for Measuring Al-Ni Diffusion Thickness on End Bonded HDS Fuel Elements

FIGURE 25

Aluminum-Nickel Diffusion Thickness on the Base of HDS Fuel Elements as Influenced by Test Variables for the Differential Thermal Expansion End Bond Test.

FIGURE 26

Aluminum-Nickel Diffusion Thickness on the Cap End of HDS Fuel Elements as Influenced by Test Variables for the Differential Thermal Expansion End Bond Test

FIGURE 27

Predicted Response Surface of Al-Ni Diffusion Thickness at Points 5/8 in. and 1-1/2 in. from the Base End (ID₁₀ and ID₁₈, Respectively,) on the Inner Surface of HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies.

FIGURE 28

Predicted Response Surfaces of Al-Ni Diffusion Thickness at Points 5/8 in. and 1-1/2 in. from the Cap End (ID₁₀ and ID₁₈, Respectively) on the Inner Surface of HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies.

FIGURE 29

Predicted Al-Ni Diffusion Thickness 5/8 in. from the Base-End on the Outside of HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies.

FIGURE 30

Predicted Al-Ni Diffusion Thickness 1-1/2 in. from the Base End on the Outside of HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies.

FIGURE 31

Predicted Al-Ni Diffusion Thickness 5/8 in. from the Cap End on the Outside of HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies

FIGURE 32

Predicted Al-Ni Diffusion Thickness 1-1/2 in. from the Cap End on the Outside of HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies.

points OD₁ and OD₂, which are 5/8 and 1-1/2 in. from the ends, respectively, are shown separately. For both the cap and base ends, time had the most pronounced effect. There was not as great a difference between points 5/8 in. and 1-1/2 in. from the ends as there was for corresponding internal bond locations. Temperature differences between the two points on the outside were not as large as they were on the inside of the piece.

Grading consisted of estimating the quantities of four types or degrees of bonding. The four types were defined as follows and are illustrated in Figure 33.

Diffusion Bond Types

<u>Type No.</u>	<u>Description</u>
1	No contact between aluminum jacket and nickel plate.
2	Intimate contact between aluminum jacket and nickel plate, but no observable diffusion at 400°C.
3	Slight diffusion observed at 400°C appearing as a single layer.
4	Gross diffusion characterized by double layer, both Al ₃ Ni ₂ and Al ₃ Ni compounds formed.

Longer times and higher temperatures produce higher percentages of type 4 bonding. The effects of pressure were not clear cut and results indicate that there may be a location-pressure interaction.⁽¹⁶⁾ The bulk of bond type percentages were type 3 and 4 except for the cap end where the most percentages were bond types 2 and 4. Variation in percentage of bond types between the cap end and base end was due to the degree of nickel oxidation during and after sizing. Larger amounts of nickel oxide were produced on the cap end by air drawn into the fuel assembly when the piece was chilled. Contact with the dies and ram tips chills the assembly, creating a slight vacuum within the fuel assembly drawing air into the can. Because a heavier oxide is produced at the cap end, it is more difficult to penetrate; however, if penetration does occur, diffusion proceeds as rapidly as it did at the base end. Due to variation in oxide thickness and penetration, the resulting diffusion bonds vary. In regions where the oxide is heavy, no diffusion takes place, resulting in type 2 bonds. However, in areas where oxide penetration occurs, normal diffusion for time-temperature conditions was noted. Distribution of bond types for the various locations, shown in Figure 23, on fuel element cap and base ends are shown in the following table.

Percentages of Bond Type

<u>Location (See Figures 23 and 24)</u>		<u>Percentage of Bond Type</u>			
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Base Section A	End	0.7	7.9	33.4	57.5
Points	1-10				
Cap Section E	End	5.7	56.5	9.8	28.0
Points	1-10				

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Type 1
No Contact

Type 2
Contact - But no
Observable
Diffusion

Type 3
Slight
Diffusion
Observable

Type 4
Gross Diffusion
Characterized
by Obvious
Double Layer

(All 500X)

FIGURE 33

Aluminum-Nickel Diffusion Bond Types used for Grading

Location (See Figures 23 and 24)		Percentage of Bond Type			
		1	2	3	4
Base Section A	Internal	0.5	3.7	39.5	56.2
Cap Section E	Internal	1.0	49.2	19.5	29.7
Base Section A	External	0.5	2.2	39.0	58.0
Cap Section E	External	0.0	28.7	21.2	49.0
Base. B Points	Internal 10-18	0.0	10.0	40.5	49.4
Cap D Points	Internal 10-18	0.0	10.1	28.1	61.2
Base B Points	External 1-9	0.0	10.1	38.3	52.2
Cap D Points	External 1-9	0.0	13.9	26.0	60.2

The effects of process variables on the formation of bond types 1, 2, 3, and 4, along with types 3 and 4 combined, are shown in Figure 34. The effect of process variables on each of the types is directly influenced by the production of another type since the total percentage of all four types on a piece has to equal 100 per cent. For example, on the base end as time increases, type 3 percentages decrease and type 4 percentages increase. Although the type 1 bond is undesirable, the type 1 chart can be read to give the amounts of other bond types formed. By subtracting the indicated percentage of type 1 bond from 100, the percentages of types 2, 3, and 4 combined can be determined. The formation of bond types 2, 3, and 4 is evolutionary in nature; i.e., type 2 changes to 3 which in turn converts to 4. Bond type 3 consists mainly of Al_3Ni with a very thin, dark layer of Al_3Ni_2 as observed at 400X. Type 4 bonding is actually an advanced state of type 3, but is much more pronounced and easily recognized.

Bond types 3 and 4 were combined and plotted on the same chart with the four other types. Bond type 2 was excluded from this part of the analysis since a possibility of intimate contact but no diffusion actually existed.

A magnification of 400X is not sufficiently high to determine whether or not diffusion had occurred. However, a magnification of 400X was sufficient to determine the effects of process variables on Al-Ni diffusion. Note that none of the process variables affected the formation of both bond types 3 and 4 at the base end when they were combined. However, at the cap end, as time and temperature increase, formation of the combination of types 3 and 4 increase. Percentages of types 3 and 4 combined at the cap end are not as high as at the base end. However, at higher cap end bonding temperatures and longer times, the amounts of type 3 and 4 bonds approach quantities produced at the base end.

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FIGURE 34

The Effects of Test Variables upon Al-Ni Diffusion on HDS Fuel Elements End Bonded with Differential Thermal Expansion Dies. (For Bond Types see Figure 33)

During evaluation of pieces end bonded for this test, large grains in the can wall were detected. However, these large grains were not considered detrimental to fuel quality. Grain etching with a macro-etch brought out bands of large grains near the base and the cap. An example of large grain distribution is shown in Figure 35. The grains were produced in layers and were believed to have been caused by a combination of stresses set up within the aluminum can during impacting and working during sizing. Heating the piece during end bonding then caused the grains to grow in layers following flow lines within the can. Cross sections of cap and base end large grain formations are shown in Figure 36.

Several tests for reducing large grain formation were tried, and should the need arise, there are methods which could be used to control can wall grain size. Annealing the can prior to sizing, reducing wall reduction and modifying the can base all reduced grain size. Can base modification would have to be made at the time of impacting because residual stresses were still evident in cans modified by machining. Modification of the core end could also help reduce grain size.

The presence of large grain formation in the can wall was not considered to be detrimental to fuel quality for a number of reasons which are listed as follows:

- 1) Large grains have not been known to have a detrimental effect upon the corrosion resistance of either C-64 or 8001 aluminum alloys.
- 2) Hot press fuel having large grains have been successfully irradiated at SRP.
- 3) The grain formation was in layers, one grain upon another, and did not consist of single large grains.
- 4) Nickel distribution throughout the large grain areas was normal.

Conclusions and Recommendations--Differential Thermal Expansion Die Test

After evaluation of data generated for the differential thermal expansion die test was completed, the following end bonding process conditions for hot die sized fuel elements were recommended:

Supplemental Preheat (HDS Fuel Assembled with Dies)	5 Minutes
Press Head Temperature	640 C
Pressure	4 T/in. ²
Press Time	4 Minutes

These conditions were chosen to maximize end bonding at the cap end without reducing external bond strength outside the die cup to less than 10,000 psi tensile strength. Equivalent force to pull a 0.190 inch diameter stud is 294 pounds.

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FIGURE 35

Typical Large Grain Distribution on an End Bonded and Welded HDS Fuel Element

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Cap End

Base End

FIGURE 36

Cross Sections of Cap and Base Large Grain Formation on an End
Bonded and Welded HDS Fuel Element

USE OF ALSi BRAZE WAFERS TO BOND CORE ENDS

Upon completion of the differential thermal expansion test, 224 fuel elements were fabricated for the initial irradiation testing of hot die sized fuel elements. However, during autoclave testing, three of the pieces failed due to defects in the closure zone.⁽¹⁷⁾ For this reason, the decision to introduce a secondary barrier to the core on the ends and to improve the weld closure zone was made. A special piece assembled and sized with an ALSi wafer at each end was end bonded under conditions of run No. 12 of Replicate III during end bonding of the previously discussed differential thermal expansion die test (See Appendix B). Metallographic examination revealed that the piece was completely end bonded; i.e., the ALSi was bonded both to the cap and base ends and to the nickel plated core.⁽¹⁸⁾

The 0.010 in. ALSi wafer consisted of about nine per cent silicon and contained about 0.2 per cent nickel because it was rolled from ALSi taken from the ALSi brazing duplex furnace. In this case, the ALSi wafer was degreased by dipping in hot trichloroethylene. No chemical cleaning or etching was used.

Subsequent wafer testing consisted of rolling ten per cent silicon ALSi wafers from makeup metal for use in the ALSi brazing duplex furnace. The wafers were cleaned by chemical etching to remove any traces of oxide which could hinder aluminum wetting and ALSi-Ni diffusion. The wafers were cleaned as follows:

- 1) Caustic etch: Turco 4181-19, 2 oz./gal. at 65 to 70 C for 30 seconds
- 2) Water rinse
- 3) Caustic etch: Dip in No. 1 solution for 4 seconds
- 4) Nitric-hydrofluoric acid: 30% HNO₃, 0.5% HF, and 69.5% water for one minute
- 5) Nitric acid: 20% for two minutes
- 6) Water rinse
- 7) Methanol rinse
- 8) Air dry

The above wafer cleaning procedure has been used for all subsequent canning runs including the initial reactor test material, the 0.96 per cent U-235 enriched uranium test and the second natural uranium test. It is expected that a less complex cleaning procedure can be developed because of the initial good experience with merely degreasing the wafers.

During sizing, the ALSi wafers are turned up and flow between the cap and can as illustrated in Figure 37. Figure 38 illustrates a typical ALSi wafer, cap and can junction, on a piece that the wafer completely wetted. Note the typical ALSi wetted aluminum. Figure 39 shows an example of the junction between a non-melted ALSi wafer and the cap and can wall. There appears to be some evidence of solid state diffusion between the can wall and ALSi wafer.

Diffusion bonding between the ALSi and nickel is shown in Figure 40. Figure 40(a) shows diffusion between ALSi-Ni diffusion when the ALSi has reached its

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FIGURE 37

Illustration of AlSi Wafer Turning up and Flowing Between Cap and Can

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FIGURE 38

Typical AlSi Wafer, Cap, and Can Junction on HDS Fuel Element End
Bonded at High Enough Temperature to Melt AlSi ($> 577^{\circ}\text{C}$)

FIGURE 39

AlSi Wafer, Cap and Can Wall Junction on HDS Fuel Element when end Bonded Below AlSi Melting Temperature (<577 C)

40 (a) End Bonded above 577 C

40 (b) End Bonded below 577 C

FIGURE 40

Typical AlSi-Ni Diffusion for End Bonded HDS Fuel Elements Above and Below AlSi Melting Temperature - 577 C

melting temperature (~ 577 C). An example of AlSi-Ni diffusion, for temperatures below the AlSi melting point, is shown in Figure 40(b).

Production Test Experience

Bonding of the AlSi wafer to the cap end was of most interest in the metallography of production test material. AlSi wafers have been completely diffusion bonded to all samples examined including the first reactor test material, (19) the enriched uranium test, and the latest test being evaluated. AlSi and nickel appear to bond more readily than aluminum and nickel.

The aluminum-AlSi junction was examined to determine the degree of bonding. On fuel elements heated sufficiently to melt the AlSi, wetting of the aluminum occurred. Good mechanical contact between the cap, AlSi wafer, and core was observed in all cases.

The AlSi wafer was forced up and between the cap and the fuel lateral surface cladding. Average height the AlSi wafer was folded up between the outer and inner cladding was 0.035 inch and 0.041 inch, respectively. More wetting was observed on the can and spire tube sides of the folded AlSi wafer than on the cap side. Increased wetting on the tube clad sides was believed due to the higher temperature of the clad side and to more friction between the tubing and the AlSi than between the cap and AlSi.

Base end bonding has been more consistent than cap end bonding because there is less aluminum and nickel oxide to hinder diffusion at the base.

Examination of the one-inch section taken adjacent to the cap and base sections revealed good nickel-uranium and aluminum-nickel diffusion. No large-scale cracking was observed in the end zones under the differential thermal expansion dies in the area adjacent to the dies.

The above comments summarizing the first hot die sized production test metallography examinations are applicable to the 0.96 per cent U-235 enriched uranium test and the latest test which is about 30 per cent complete. Cap wetting for the six-inch enriched fuel test was more consistent because of increased heating rates due to less core weight. Increased heating rate was compensated for by a reduction in preheat and press times. End bonding conditions for the eight-inch natural and six-inch enriched production tests are summarized below:

	<u>Eight-Inch Fuel</u>	<u>Six-Inch Fuel</u>
Supplemental Preheat Furnace Temperature Setting	680 C	680 C
Assembly Preheat Time $\pm$ 15 Seconds	5-3/4 minutes	4-3/4 minutes
Press Head Temperature $\pm$ 5 C	655 C	655 C
Press Time $\pm$ 3 seconds*	4-1/2 minutes	4-1/4 minutes
Pressure $\pm$ 1/4 T/in. ²	4 T/in. ²	4 T/in. ²

*Press time variation for manual control was $\pm$ 10 seconds. Installation of automatic timer made tighter control possible.

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Use of the above conditions has produced hot die sized fuel elements having highly reliable end bonds. The introduction of automatic timing equipment has notably increased uniformity of quality. Additional mechanization and use of an induction heating system is expected to further increase the reliability of end bonding on hot die sized fuel. It is speculated that with additional development work, end bonding and/or closure welding steps may be eliminated. It may be practical to end bond pieces at the time of sizing. Proposed methods include (1) use of induction heating to supply additional heat immediately after sizing in the sizing press itself, (2) use of low melting braze wafers, and (3) use of modified aluminum alloy components.

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APPENDIX A

END BOND TEST

Testing sequence supplied by K. B. Stewart, Applied Mathematics Operation, Hanford Laboratories (Reference 3).

Variables to be Studied

	Test 1 Preheat 5 Min. at 630 C	Test 2 No Preheat
Press Head Temperature	560, 600, and 640 C	560, 600, and 640 C
Press Time	30, 105, and 180 Seconds	5, 7, and 9 minutes
Pressure	120, 160, and 200 psig*	120, 160, and 200 psig*

Experimental Sequence

Two pieces run at each set of conditions and each test was repeated.

Run	Temp. °C	Time (Sec.)	Pressure* psig	Run	Temp. °C	Time (Min.)	Pressure psig
1	600	30	120	1	600	5	120
2	560	105	200	2	600	9	120
3	640	30	160	3	640	9	160
4	600	105	160	4	640	7	200
5	560	30	160	5	600	7	160
6	600	180	120	6	640	7	120
7	640	180	160	7	560	9	160
8	600	30	200	8	560	7	200
9	560	105	120	9	640	5	160
10	600	105	160	10	600	7	160
11	560	180	160	11	600	5	200
12	600	180	200	12	560	7	120
13	640	105	200	13	560	5	160
14	600	105	160	14	600	7	160
15	640	105	120	15	600	9	200

*Test design shows gage pressure.

Pressure converted to T/in.² applied on end of piece.

120 psig = 1.03 T/in.²
 160 psig = 1.38 T/in.²
 200 psig = 1.72 T/in.²

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APPENDIX B

DIFFERENTIAL THERMAL EXPANSION DIE END BONDING TEST

Testing sequence supplied by K. B. Stewart, Applied Mathematics Operation, Hanford Laboratories (Reference 11).

Variables to be Studied

Press Head Temperature*	610, 640, and 670 C
Press Time	2.5, 3.5, and 4.5 minutes
Pressure	3, 4, and 5 T/in. ²

* Pieces fitted with end bond die cups and plugs preheated for 5 minutes in the resistance furnace set at 680 C.

Experimental Sequence

Three pieces end bonded for each of the following conditions:

REPLICATE I

<u>Run</u>	<u>Time Minutes</u>	<u>Temperature °C</u>	<u>Pressure T/in.²</u>
1	4.5	640	3
2	3.5	640	4
3	2.5	640	3
4	3.5	640	4
5	3.5	670	3
6	2.5	640	5
7	2.5	670	4
8	4.5	610	4
9	2.5	610	4
10	4.5	670	4
11	3.5	670	5
12	4.5	640	5
13	3.5	610	5
14	3.5	610	3
15	3.5	640	4

REPLICATE II

<u>Run</u>	<u>Time Minutes</u>	<u>Temperature °C</u>	<u>Pressure T/in.²</u>
1	4.5	640	3
2	3.5	640	4
3	2.5	640	5
4	2.5	640	3
5	4.5	670	4

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APPENDIX B (Cont'd)

Replicate II (Cont'd)

<u>Run</u>	<u>Time Minutes</u>	<u>Temperature °C</u>	<u>Pressure T/in.²</u>
6	2.5	610	4
7	3.5	610	5
8	4.5	610	4
9	3.5	610	3
10	3.5	640	4
11	3.5	670	3
12	3.5	670	5
13	2.5	670	4
14	4.5	640	5
15	3.5	640	4

REPLICATE III

<u>Run</u>	<u>Time Minutes</u>	<u>Temperature °C</u>	<u>Pressure T/in.²</u>
1	2.5	640	3
2	2.5	610	4
3	4.5	610	4
4	3.5	640	4
5	3.5	670	3
6	3.5	670	5
7	3.5	610	3
8	4.5	640	5
9	3.5	640	4
10	3.5	610	5
11	4.5	640	3
12	4.5	670	4
13	3.5	640	4
14	2.5	670	4
15	2.5	640	5

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