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GENERAL ELECTRIC

HANFORD ATOMIC PRODUCTS OPERATION - RICHLAND, WASHINGTON

May 22, 1963

TITLE

INTERIM REPORT ON
HOT DIE SIZING VARIABLES TEST

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INTERIM REPORT ON
HOT DIE SIZING VARIABLES TEST

May 20, 1963

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Process Development Unit
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Production Fuels Section, IPD

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INTRODUCTION

Studies were initiated at Hanford in 1961 and 1962 toward the development of an alternate assembly process for the production of I&E fuel elements for the eight existing Hanford reactors. Of the processes considered, hot die sizing, a diffusion bonding process, offered the greatest incentives in terms of improved quality and potentially cheaper unit cost of fuel elements compared to the currently used AlSi braze process. This interim report presents the results of initial process variables tests designed to establish optimum process parameters for producing good diffusion bonds on the lateral external and internal surfaces of I&E fuel elements during the die sizing step of the hot die size process. In a subsequent step, the end bonds are formed. Optimization studies for producing good end bonds will be reported in future interim reports.

SUMMARY

Four parameters were selected for optimization of the die sizing step. The four parameters and the ranges which were investigated were:

Preheat furnace temperature, °C	650, 680, 710
Preheat furnace time, minutes	8-1/2, 10, 11-1/2
Can wall reduction, per cent	27.3, 45.5, 63.5
Ram speed, in. per minute	40, 65, 90

Results of statistical analysis of non-destructive and destructive examination of fuel elements sized by the above conditions has led to the following conclusions:

- 1) None of the process variables influenced internal and external total bond count significantly.
- 2) Except for wall reduction, variation in external dimensions were mainly due to piece temperature at the time of sizing. Controlling preheat temperature and time would aid in producing uniform pieces. In general the higher the piece temperature at the time of sizing, the smaller the external diameter of the finished piece.
- 3) Internal diameter was controlled by wall reduction, but slightly influenced by ram speed and preheat temperature.
- 4) Variation in external diameter along the piece was also a function of piece temperature during sizing--usually the hotter the piece the smaller the diameter. Use of insulated ram tips and controlled temperature dies should help reduce the variation. There was some variation in external diameter at the cap end due to ram speed which was believed to have been caused by variation in ram coasting after activating the stop mechanism for the different ram speeds.

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- 5) Wall reduction was the major variable affecting internal clad thickness. Other variables affecting the over-all thickness include ram speed and preheat furnace temperature.
- 6) In addition to wall reduction, preheat time and temperature influenced external clad thickness. The longer the time and higher the temperature, the thinner the external clad thickness.
- 7) External bond strength at the cap end of hot die sized fuel elements will have to be improved. Test data indicate that increasing wall reduction should increase the bond strength. For this reason, test quantities of thick wall components have been ordered. Increased wall reduction can be tested using existing external dies. Recent tests using an improved argon purge also appear to have improved cap end bonding. Improved inert gas shielding was achieved by pulling a vacuum on assembled pieces and then back filling with argon. The vacuum dryer has now been modified to use this back filling technique.
- 8) Internal bond strength was also lowest at the cap end. Use of improved inert gas shielding is expected to aid in this area.
- 9) Aluminum-nickel diffusion thickness increased as piece temperature increased. In the case of the internal surface, greater wall reduction also caused an increase in diffusion thickness at higher temperatures.
- 10) Internal and external wall thickness was mainly a function of wall reduction; however, external clad thickness was also affected by piece temperature.
- 11) Tentative process conditions for producing hot die sized fuel elements are:
 - a) Preheat furnace temperature 680 ± 10 C
 - b) Preheat furnace time 10.0 ± 0.5 minutes*
 - c) Wall reduction ~ 55 per cent**
 - d) Ram speed 70 ± 5 inches per minute

*Changed to 12.0 ± 0.5 min. after circulating fan removed from furnace.

**To give the desired finished dimensions, a 36 per cent to 45 per cent wall reduction will be required. It would be desirable to increase wall reduction, but is impossible to achieve the desired finished dimensions until thicker wall components are available.

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DISCUSSION

Hot die sizing (HDS) consists of passing a preheated core-component fuel assembly through a cold die to form the external lateral bond and in the same operation, passing a mandrel through the internal bore of the assembly to form the internal lateral bond. Subsequently, in another step, the end bonds are formed.

Test variables studied are listed below:

Test Variables

Preheat furnace temperature	650, 680, and 710 C
Preheat furnace time	8-1/2, 10, and 11-1/2 min.
Can wall reduction(WR)*	27.3, 45.5, and 63.5 per cent
Ram speed	40, 65, and 90 in./min.

$$*WR \text{ defined as } \frac{(100)(W_1 - W_2)}{W_1}$$

Where W_1 = Nominal can wall thickness before sizing
 W_2 = Nominal can wall thickness after sizing

This test was divided into six blocks, each of which was to be run on a separate day. Each block (day) consisted of nine separate runs of four pieces each. (For a more detailed description of the test, see Appendix A.)

Nickel Plating

Dingot uranium cores which had been vacuum precipitation annealed were used for the test. Vacuum annealing consisted of holding the pieces at 1000 F for 16 hours in a vacuum furnace prior to plating for the purpose of precipitating the carbon in the uranium cores to produce a structure more similar to ingot uranium. During the time pieces were being prepared for HDS testing, considerable work was being done in the area of improving the performance of dingot uranium fuel elements. Vacuum precipitation annealing was one of the improvement methods suggested by the feed site representatives. The cores were then plated in a Watt's plating bath which is described in detail in HW-77759, pp. 108-117.

Aluminum Components and Cleaning

Components for hot die sized fuel elements consisted of an integral spire can and a cap wafer fabricated according to drawing numbers H-3-17365, Rev. 1 and H-3-17364, respectively. Cap wafer diameters were modified to prevent upsetting and seizing the flash during sizing. Inner and outer diameters were changed to 0.453 in. and 1.400 in., respectively. The components were cleaned by etching one minute in an inhibited caustic etch, 5 oz. Aluminux per gallon of solution at 30 to 35 C followed by

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a hot water rinse and a quick dip in the Aluminux etch (less than 10 seconds) to remove miscellaneous oxidation and reaction products. The pieces were then immediately rinsed in hot and cold water and then deoxidized in a 32 oz. per gal. Diversey 514 solution for five minutes at room temperature. The deoxidation step was followed by two water rinses and two methanol dips. The components were then placed in the vacuum dryer and held until needed.

Component Assembly

Immediately prior to assembly, the aluminum cans were purged with argon to reduce nickel and aluminum oxidation during preheat. After assembly, the pieces were dipped in a solution containing colloidal graphite (one part aqua-dag to four parts water) for lubrication during sizing. The aqua-dag suspension adhered to aluminum components roughened by a caustic etch much better than to components cleaned by standard cleaning solutions which do not etch the surface. After drying, the fuel assembly was placed in the heat shield as illustrated in Figure 1 showing components, dies, an as-sized piece and a finished hot die sized fuel element. Heat shields serve two purposes: (1) to maintain fuel heat while being transferred from the preheat furnace to the press and (2) to align the piece in the press.

Fuel Preheat

Fuel assemblies in heat shields were preheated according to test conditions in a resistance furnace. Test preheat conditions were established after testing conditions encompassing a wide range of time and temperature known to be satisfactory for sizing. Uranium core temperatures were obtained by embedding thermocouples at the ends and center of fuel cores. Typical uranium core temperature curves are shown in Figures 2 and 3 with and without the circulating fan, respectively. Note that higher core temperatures were obtained by use of a circulating fan in the preheat furnace. Early work indicated that it was highly desirable to heat the cores as rapidly as possible to prevent uranium oxidation and associated damage to the nickel plate. Since then, nickel plate quality has been improved to the extent that preheat furnace time does not seriously affect nickel plate quality. The preheat furnace fan has been removed due to a maintenance problem caused by overheating the fan shaft bearings.

Sizing

Hot die sizing of I&E fuel elements consists of sizing preheated fuel assemblies with a 50 ton Hannifin press equipped with a specially adapted lower ram assembly to supply back pressure as the piece is being sized. The lower ram is bored to accommodate the support stem which holds the inner die. Figure 4 illustrates the sizing process to show the relationship between the dies, fuel assembly, and upper and lower rams.

Prior to each sizing run the press was prepared to give the prescribed wall reduction and ram speed. The desired wall reduction was obtained by equipping the press with the proper inner and outer dies. Inner and outer dies were fabricated from tool steel and are described in Drawings H-3-17424

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FIGURE 1

Display of Materials used to Make Hot Die Sized Fuel Elements. Items from Left to Right are:
Nickel Plated Core, Cap and Can, Can-Core Assembly in Opened Heat Shield, Internal Die,
External Die Assembly, As-Sized Piece and a Finished Hot Die Sized Fuel Element.

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FIGURE 2 - URANIUM CORE TEMPERATURES DURING FUEL ASSEMBLY PREHEAT WITH THE CIRCULATING FAN ON.

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FIGURE 3 - URANIUM CORE TEMPERATURES DURING FUEL ASSEMBLY PREHEAT WITHOUT THE CIRCULATING FAN.

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FIGURE 4 - HOT DIE SIZING PROCESS

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and H-3-17425, respectively. Ram speed was controlled by adjusting the press hydraulic unit oil flow rate and was measured by a timer attached to the press.

Die lubrication consisted of brushing the inner and outer dies with warm Fiske No. 604 extrusion lubricant. The combination of lubricated dies and aqua-dag coated components produced the best surface after sizing with tool steel dies. Other lubricating materials tested consisted of various dilutions of Fiske No. 604 with motor oil, mixtures of Fiske 604 with aqua-dag, etc. During initial testing it was observed that fuel surface appearance was improved by preheating the outer die and case. Apparently a cold die picks up more aluminum than a warm one, making it necessary to clean the die oftener. During the optimization test, dies were cleaned by dipping in a 50 per cent caustic solution whenever there was an aluminum build-up on the dies.

After each piece was sized, the upper ram tip was changed to prevent cap wafer upsetting. Cap upsetting caused seizure of the inner and outer can wall, putting considerable stress on the inner die support rod. Whenever a cap upset and external clad wrinkling was observed, the sizing press was stopped immediately to prevent damage to the inner die support. Immediately after sizing, the inner die was removed manually by forcing it up through the inner flash with a thin rod pushed through the fuel element. If this operation were done soon enough after sizing, the die could be removed quite easily. For this reason, a piece of equipment has been designed and built to remove the dies. After removing the inner dies, pieces were set aside to air cool.

Facing and Cleaning

When all the pieces for the test were assembled, the inner and outer flash at the top of each piece was faced off and the sharp corners were chamfered to aid in testing. Faced pieces were cleaned by soaking in trichloroethylene and scrubbing with acetone. It was obvious after starting to clean the pieces that pieces cleaned immediately after sizing were easier to clean. Later tests have indicated that water quenching immediately after sizing caused most of the baked on Fiske No. 604 and aqua-dag to be blasted off by steam formation.

Testing and Evaluation of Results

Evaluation of test pieces consisted of subjecting the pieces to non-destructive testing followed by destructive testing. Nondestructive and destructive tests are itemized as follows:

Nondestructive Tests

1. Ultrasonic bond count - Internal
2. Ultrasonic bond count - External
3. Internal dimensions - 2 places
4. External dimensions - 5 places
5. Clad thickness test

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Destructive Tests

1. External bond strength
2. Internal bond strength
3. Metallography - Al-Ni and Ni-U diffusion
4. Clad thickness - O.D. and I.D.
5. Clad thickness in closure zone.
6. Base end non-bonding
7. Cap end non-bonding

All data taken were analyzed by Applied Mathematics and the results were evaluated for each of the tests made. In most cases, one or more of the process variables influenced the test and whenever possible the results are shown in a three-dimensional response surface to simplify the discussion. Formulas for each of the response surfaces are given in the Appendix.

Ultrasonic Bond Count

Results of the ultrasonic bond test were inconclusive. At first there appeared to be a correlation between total external bond count and wall reduction; however, it was later determined that the tester was responding to galling on the fuel surface. During early testing there was more galling on pieces sized at the higher wall reductions. None of the process variables influenced the internal total bond count.

Dimensions

The inside and outside diameters of hot die sized fuel elements produced for the optimization test were measured in two and five places, respectively. These locations were chosen as a result of measurements taken on pieces during early sizing tests. It was noted that the end external diameters were large compared to diameters taken at the center region (center portion 1-1/2 inches from each end). Internal diameters did not vary much except in the immediate end zone. For these reasons, internal and external diameters were taken at the locations shown in Figure 5. Graphs showing internal and external diameter measurements taken at 1/2 inch intervals on hot-die sized pieces are shown in Figures 6 and 7. It has been concluded that variation in diameter along the uranium core is due to the difference in temperature along the piece during sizing. Large diameters at the ends were due to end cap upsetting during sizing. Internal diameter measurements indicated that smaller external diameters were obtained as the core temperature increased because of the difference in core size during sizing. Core end diameters are larger because the upper and lower rams conduct heat away from the core ends.

Wall reduction was the only variable that influenced external diameter measurements 1/2 inch from the base end (O.D.-1) as shown in Figure 8. All variables with the exception of ram speed affected O.D.-2, O.D.-3, and O.D.-4. In general, increased preheat time and increased preheat furnace temperature as well as increased wall reduction caused a reduction in these three diameters. A comparison of the formulas for each of the three diameters (O.D.-2, O.D.-3, and O.D.-4) revealed very little

FIGURE 5 - LOCATION OF INTERNAL AND EXTERNAL DIAMETER MEASUREMENTS FOR PIECES ASSEMBLED FOR THE HOT DIE SIZING OPTIMIZATION TEST.

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FIGURE 6 - INTERNAL DIAMETERS TAKEN AT 1/2 IN. INTERVALS ON THREE HOT DIE SIZED FUEL ELEMENTS.
PROCESS CONDITIONS: ASSEMBLY PREHEATED FOR 9 MINUTES AT 700 C AND SIZED WITH A NO. 3 INTERNAL TOOL STEEL DIE.

FIGURE 7 - EXTERNAL DIAMETERS TAKEN AT 1/2 IN. INTERVALS ON THREE HOT DIE SIZED FUEL ELEMENTS.
PROCESS CONDITIONS: ASSEMBLY PREHEATED FOR 9 MINUTES AT 700 C AND SIZED WITH A NO. 6 TOOL STEEL DIE.

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FIGURE 8 - PREDICTED RESPONSE SURFACES FOR EXTERNAL DIAMETERS 1/2" FROM THE BASE END (OD-1) FOR FUEL ELEMENTS PRODUCED FOR THE HOT DIE SIZING OPTIMIZATION TEST SHOWING THE EFFECT OF TEST VARIABLES.

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difference in variable effects when compared to measurement capability. The three formulas were averaged to give the response surface shown in Figure 9. (See Formula 5 in Appendix B.) Cap-end diameter (O.D.-5), which is illustrated in Figure 10, was influenced by all process variables and increased as: (1) wall reduction decreased, (2) furnace temperature decreased, (3) preheat time increased, and (4) ram speed decreased at short preheat time and as ram speed increased at a long preheat time.

Wall reduction was the most dominant variable observed on the internal diameter measurements. In the case of diameters 1-1/2 inch from the base (I.D.-1), both ram speed and an interaction between preheat temperature and wall reduction significantly influenced the diameter while an interaction between ram speed and wall reduction influenced internal diameter measurements 1-1/2 inches from the cap (I.D.-2). Note that in Figure 11 internal diameters became smaller as preheat temperature increased when a low wall reduction was used; however, the opposite was true for the intermediate and high wall reduction. The lowest ram speed produced the largest internal diameters for low wall reduction. Ram speed did not significantly affect diameters for the intermediate level of wall reduction. The effect of process variables upon I.D.-2 dimensions are shown in Figure 12.

External Clad Thickness Test

Analysis of data taken on two replicates of the test revealed that the eddy current clad thickness tester will be a useful production tool. Since the clad thickness tester was in the development stage, only one-third of the test pieces were tested. Results showed that higher wall reductions produced thinner wall thickness and that longer preheats resulted in thinner cladding. The data were in good agreement with external clad thickness measurements obtained by destructive testing.

External Bond Strength

External bond strength measurements on hot die sized fuel elements were made by stud welding twelve 0.25 inch 6061-T6 aluminum alloy studs to each piece at points 1-1/4 inches from each end and at the center in four rows about 90 degrees apart. External bond strengths are expressed as pounds force to pull the 0.25 inch stud rather than converting to tensile strength. During the test, the size of the can wall button removed with the stud varied, and it was apparent that converting pounds force to tensile strength would not be expressing true bond strength.

Predicted bond strengths for the three locations, cap, center, and base, are illustrated in response surface diagrams shown in Figure 13. It will be noted that wall reduction was the only process variable significantly affecting bond strength 1-1/4 inch from the cap and that cap-end bond strengths were lower than those measured at the other locations. Preheat furnace time and temperature as well as reduction ratio affected bond strength at the center of hot die sized pieces while none of the variables affected bond strength 1-1/4 inch from the base end. Of primary interest was the effect of wall reduction on bond strength at the cap end. It is

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FIGURE 9 - PREDICTED RESPONSE SURFACES FOR DIAMETERS IN THE CENTRAL ZONE OF FUEL ELEMENTS PRODUCED FOR THE HOT DIE SIZING OPTIMIZATION TEST. MEASUREMENT LOCATION: 1-1/2 IN. FROM THE BASE, CENTER AND 1-1/2 IN. FROM THE CAP (OD-2, OD-3, AND OD-4).

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FIGURE 10 - PREDICTED RESPONSE SURFACES FOR DIAMETERS 1/2 IN. FROM THE CAP END (OD-5) OF FUEL ELEMENTS PRODUCED FOR THE HOT DIE SIZING OPTIMIZATION TEST SHOWING THE EFFECTS OF PROCESS VARIABLES.

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FIGURE 11 - PREDICTED RESPONSE SURFACES SHOWING THE EFFECTS OF PROCESS VARIABLES UPON INTERNAL DIAMETERS 1-1/2 IN. FROM THE BASE (ID-1) ON FUEL ELEMENTS PRODUCED FOR THE HOT DIE SIZING OPTIMIZATION TEST.

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FIGURE 12 - PREDICTED RESPONSE SURFACES SHOWING THE EFFECT OF PROCESS VARIABLES UPON INTERNAL DIAMETERS 1-1/2 IN. FROM THE CAP (ID-2) ON FUEL ELEMENTS PRODUCED FOR THE HOT DIE SIZING OPTIMIZATION TEST.

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FIGURE 13 - PREDICTED RESPONSE SURFACES SHOWING THE EFFECTS OF PROCESS VARIABLES UPON EXTERNAL BOND STRENGTH AT POINTS 1-1/4 IN. FROM THE CAP, CENTER, AND 1 1/4 IN. FROM THE BASE

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theorized that as wall reduction is increased, more positive smearing action between the aluminum can and nickel plate is accomplished, causing a breaking up of oxide layers which could be preventing aluminum-nickel diffusion from taking place. For this reason, additional components having thicker can walls will be tested at higher wall reductions to further improve cap end bonding.

It has also been determined more recently that the amount of oxidation taking place before, during, and after sizing also influence cap end bonding. Improved argon purging and tight fitting cap wafers have been demonstrated to increase cap end bond strength.

Internal Bond Strength

Internal bond strength measurements were taken on only one of the pieces for each of the hot die sizing test conditions because of the time required for splitting the pieces lengthwise and the large number of cores destroyed. Because of the small radius on the fuel element inner surface, it was necessary to grind off two opposite edges of 0.190 inch diameter studs. Bond strength was measured, 1-1/4 inch from each end and in the center. None of the process variables affected the bond strength 1-1/4 inch from the cap end significantly. Preheat furnace temperature was the major process variable affecting internal bond strength at the center and 1-1/4 inch from the base. Bond strength increased as preheat temperature increased. As wall reduction increased, bond strength at the center decreased. Predicted response surfaces for bond strengths 1-1/4 inch from the cap, at the center, and 1-1/4 inch from the base are shown in Figure 14. Note that only preheat furnace temperature affected bond strength at the base end.

Metallography

Metallography of samples prepared prior to this test revealed that nickel-uranium diffusion occurred in the preheat furnace and that aluminum-nickel diffusion was initiated at the instant the aluminum jacket contacted the nickel plate during sizing. Both nickel-uranium and aluminum-nickel diffusion continue until the piece temperature drops below a critical temperature.

One piece from each of the 54 tests was sent to the Quality Control Analytical Laboratory for sectioning and polishing to permit study of the continuity and properties of the diffusion bond. Each piece was sectioned as indicated in Figure 15.

Soon after polishing began, it was apparent that obtaining a perfectly flat surface across the aluminum-nickel-uranium bond was very difficult. Due to the difference in hardness and the massive sample, it was difficult to photograph typical diffusion bonds at magnifications high enough to see the aluminum-nickel and nickel-uranium diffusion. Although it was necessary to focus the microscope constantly, it was possible to make optical observations on the test pieces. In order to obtain photomicrographs of the diffusion layers, it was necessary to cut small sections

FIGURE 14 PREDICTED RESPONSE SURFACES SHOWING THE EFFECT OF PROCESS VARIABLES UPON INTERNAL BOND STRENGTH AT LOCATIONS 1-1/4 IN. FROM THE CAP, THE CENTER, AND 1-1/4 IN FROM THE BASE.

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Longitudinal Sections Through Center of A, C, and E
Transverse Sections of B and D

FIGURE 15 - IDENTIFICATION OF SECTIONS TAKEN FOR METALLOGRAPHIC EXAMINATION OF FUEL ELEMENTS PRODUCED FOR THE HOT DIE SIZING OPTIMIZATION TEST.

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from the large pieces to obtain flat surfaces suitable for photographing at magnifications over 400X. In general, it was noted that both nickel-uranium and aluminum-nickel diffusion thickness increased as preheat time and temperature increased. Diffusion bonds for hot die sized pieces differed from those obtained by hot pressing in that diffusion thickness was less and that there was usually only one aluminum-nickel diffusion product visible on hot die sized pieces compared to two for hot pressing. Typical external hot die sizing diffusion bonds for the test temperature extremes of 650 C and 710 C after preheating for 11-1/2 minutes each are shown in Figure 16. Note the increased diffusion for the high temperature sample. Individual diffusion layers are identified in Figure 17, which is a higher magnification of the sample shown in Figure 16 (B). Identification is based upon work done by Angerman.⁽¹⁾

A typical photomicrograph of a hot press diffusion bond is shown in Figure 18. Note that the diffusion product is quite thick in spite of the low magnification (250X).

Metallographic samples were prepared for examination by etching for three seconds at five volts in a chromic-acetic acid solution.⁽²⁾ This technique was used to darken the NiU₆ layer making it more easily detected. The Ni₅U layer remained quite light, but could be seen. The etchant was prepared by diluting a stock solution consisting of 118 gm chromium trioxide per 100 mil water at a ratio of one part stock solution to four parts acetic acid.

To further study the effects of process variables upon diffusion bonding, samples of hot die sized pieces were submitted to the Uranium Metallurgy Operation, Hanford Laboratories, for electron microscopy work. Wall reduction had been observed to influence bond strength, but no difference in diffusion bond structure could be detected by optical microscopy. For this reason, samples from day No. 2 produced at the various wall reductions and temperatures, but having the same ram speed and preheat time, were chosen for examination. Two examples of aluminum-nickel and nickel-uranium diffusion bonds taken from the internal surfaces of test pieces are shown in Figures 19 and 20, respectively. Aside from the fact that the aluminum-nickel diffusion is thicker and also consists of two layers for the higher temperature test conditions, no other conclusions can be made from the electron microscopy work. Electron microscopy on the remaining samples is continuing.

Aluminum-nickel diffusion layer thickness was made on the external and internal surfaces of test pieces with a Filar eyepiece at 400X. Statistical analysis of data taken on aluminum-nickel diffusion thickness revealed that as both preheat time and preheat temperature increased, Al-Ni diffusion thickness increased. In the case of the internal Al-Ni diffusion thickness, increasing wall reduction also promoted increasing thickness at the higher preheat temperature. Predicted Al-Ni diffusion thickness measurements, based upon the optimization test for the internal and external surfaces, are shown in Figures 21 and 22, respectively. Note that wall reduction had no effect on external Al-Ni diffusion thickness.

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Negative No. 6311-OD-1-1

1000X

(A) Preheat: Temperature - 650 C, Time 11-1/2 Min.

Negative No. 6313-OD-1-1

1000X

(B) Preheat: Temperature 710 C, Time 11-1/2 Min.

FIGURE 16

Comparison of Typical External Aluminum-Nickel and Nickel-Uranium Diffusion Produced by Hot Die Sizing After Preheating for 11-1/2 Min. at 650 C and 710 C. Note the Increased Diffusion Thickness for the Higher Temperature Piece.

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Negative No. 6313-OD-1-2

1700X

FIGURE 17

Identification of Diffusion Products Produced During Hot Die Sizing.
Test Conditions: Preheat 710 C for 11-1/2 Minutes and Sized with a
45.5% Wall Reduction Die.

A Typical Hot Press Diffusion Bond

FIGURE 18

Negative No. 6403-3

250X

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Negative No. 1583 B

10,000X

(A) Preheat 10 Min. at 710 C - Wall Reduction 27.3%

Negative No. 1575 B

10,000X

(B) Preheat 10 Min at 680 C - Wall Reduction 45.5%

FIGURE 19

Examples of Internal Aluminum-Nickel Diffusion as Observed by
Electron Microscopy

Examples of Internal Nickel-Uranium Diffusion as Observed by Electron Microscopy

FIGURE 20

Negative No. 1574 D
(B) Preheat 10 Min. at 680 C Wall Reduction 45.5%
10,000X

Negative No. 1582 D
(A) Preheat - 10 Min. at 710 C Wall Reduction 27.3%
10,000X

Ni-U Diffusion
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FIGURE 21 - PREDICTED RESPONSE SURFACES SHOWING THE EFFECTS OF PROCESS VARIABLES UPON INTERNAL ALUMINUM-NICKEL DIFFUSION THICKNESS AT THE CENTER OF HOT DIE SIZED FUEL ELEMENTS.

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FIGURE 22 - PREDICTED RESPONSE SURFACES SHOWING THE EFFECTS OF PROCESS VARIABLES UPON EXTERNAL ALUMINUM-NICKEL DIFFUSION THICKNESS AT THE CENTER OF HOT DIE SIZED FUEL ELEMENTS. WALL REDUCTION HAD NO EFFECT

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Attempts to measure the nickel-uranium diffusion layers for all the pieces were unsuccessful because they were so very thin. Uranium-nickel diffusion thickness was estimated to be from 0.02 to 0.06 mils (0.5 to 1.5 microns) for the test.

Aluminum Cladding Thickness

Aluminum cladding thickness was measured at a point 1-1/4 inch from the base end, Section "B" as shown in Figure 15. Wall reduction was the only process variable that significantly affected internal clad thickness. The higher the wall reduction, the thinner the cladding. External clad thickness was similar to internal clad thickness, as would be expected for wall reduction. However, preheat time also influenced external cladding thickness. As preheat time increased, external clad thickness decreased. Predicted response surfaces for internal and external cladding thickness are shown in Figures 23 and 24, respectively.

Closure Zone Integrity

Examination of the closure zone, Section "E" of Figure 15, revealed that in some instances good bonding between the cap wafer and the lateral surface cladding occurred. For this reason, each of the pieces sectioned were examined for closure integrity and graded. However, none of the process variables influenced the degree of bonding between the cap and the inner and outer cladding significantly. Additional testing has indicated that improved bonding in this region can be obtained by use of tighter fitting cap wafers. The good bonds experienced on some of the pieces in the test were believed to have resulted from an off-center situation allowing the cap wafer to be closer to one side than the other.

Clad thickness in the closure zone was also measured on each of the same pieces. Thickness measurements were made on the inner and outer surfaces about 1/16 inch above and below the cap-core junction to compare the clad thickness at these points. As can be seen in Figures 25 and 26, clad thickness over the cap wafer was greater than over the core. This greater thickness over the cap resulted from can wall filling to the undersize cap wafer when the cap section was pushed through the die.

Cap and Base End Non-Bonding

A number of the pieces sectioned were observed to have large cracks between the end and the core; however, after examining the base and cap from each piece sectioned, Sections "A" and "E", Figure 15, the degree of cracking could not be associated with any of the process variables.

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FIGURE 23 - PREDICTED RESPONSE SURFACES SHOWING THE EFFECTS OF PROCESS VARIABLES UPON INTERNAL CLAD THICKNESS ON HOT DIE SIZED FUEL ELEMENTS.

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FIGURE 24 PREDICTED RESPONSE SURFACES SHOWING THE EFFECTS OF PROCESS VARIABLES UPON EXTERNAL CLAD THICKNESS ON HOT DIE SIZED FUEL ELEMENTS.

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FIGURE 25 : THE EFFECT OF WALL REDUCTION UPON INTERNAL CLADDING THICKNESS AT THE CLOSURE ZONE ON HOT DIE SIZED FUEL ELEMENTS.

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FIGURE 26.- THE EFFECT OF WALL REDUCTION AND PREHEAT TEMPERATURE UPON EXTERNAL CLADDING THICKNESS AT THE CLOSURE ZONE OF HOT DIE SIZED FUEL ELEMENTS.

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1. Angerman, C. L., "Metallographic Studies of Al-Ni-U Bonds in Nuclear Fuel Elements," Transactions Quarterly, September, 1961, Vol. 54, No. 3: 260-275.
2. Tews, W. G., personal communication, October 10, 1962.

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APPENDIX A

HOT DIE SIZING OPTIMIZATION TEST

Test design supplied by J. L. Jaech, Applied Mathematics, Hanford Laboratories.

Variables to be Investigated

	<u>L (low)</u>	<u>M (medium)</u>	<u>H (high)</u>
A. Preheat Furnace Temperature	650 C	680 C	710 C
B. Preheat Time (min.)	8.5	10.0	11.5
C. Ram Speed (in./min.)	40	65	90
D. Wall reduction	27.3%	45.5%	63.6%

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Appendix A (Cont'd)

Experimental Sequence

Run No.	Day 1 Treatment			
	A	B	C	D
1	M	M	L	H
2	L	L	M	M
3	M	M	H	H
4	L	H	M	M
5	H	L	M	M
6	H	H	M	M
7	M	M	L	L
8	M	M	H	L
9	M	M	M	M

(eg) Run 1 - 4 cores run at 650 C, 10 min., 40 ipm, 63.6%
WR - Can Core Code - 1-1-1, 1-1-2, 1-1-3, 1-1-4

Run No.	Day 2 Treatment			
	A	B	C	D
10	H	M	M	L
11	M	M	M	M
12	M	H	L	M
13	H	M	M	H
14	M	L	L	M
15	M	H	H	M
16	L	M	M	H
17	L	M	M	L
18	M	L	H	M

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Appendix A (Cont'd)

Run No.	Day 3 Treatment			
	A	B	C	D
19	H	M	H	M
20	M	H	M	L
21	M	L	M	H
22	H	M	L	M
23	M	H	M	H
24	M	M	M	M
25	M	L	M	L
26	L	M	H	M
27	L	M	L	M

Run No.	Day 4 Treatment			
	A	B	C	D
28	H	L	M	M
29	M	M	M	M
30	M	M	L	L
31	M	M	L	H
32	M	M	H	L
33	L	L	M	M
34	L	H	M	M
35	M	M	H	H
36	H	H	M	M

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Appendix A (Cont'd)

Run No.	Day 5			
	Treatment			
	A	B	C	D
37	M	H	L	M
38	M	L	H	M
39	M	M	M	M
40	H	H	M	H
41	H	M	M	L
42	L	M	M	H
43	M	H	H	M
44	M	L	L	M
45	L	M	M	L

Run No.	Day 6			
	Treatment			
	A	B	C	D
46	M	L	M	L
47	H	M	L	M
48	L	M	L	M
49	M	L	M	H
50	L	M	H	M
51	M	M	M	M
52	M	H	M	H
53	M	H	M	L
54	H	M	H	M

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APPENDIX B

RESPONSE SURFACE EQUATIONS

Equations for plotting the response surfaces and graphs for the hot die sizing variables test are listed below. Each equation is listed according to the order discussed in the body of the report. All equations were supplied by K. B. Stewart, Applied Mathematics Operation, Hanford Laboratories.

$$X_1 = (T-680 \text{ C})/30 \text{ C}$$

$$X_2 = (t-10 \text{ min.})/1.5 \text{ min.}$$

$$X_3 = (\text{Ram Speed}-65 \text{ ipm})/25 \text{ ipm}$$

$$X_4 = (\text{Wall Reduction}-45.5\%)/18.2\%$$

External Diameter (For locations see Figure 5) - Figures 8, 9, and 10

1. $OD_1 = 1.400 + 0.1 (0.9554 - 0.407X_4)$
2. $OD_2 = 1.400 + 0.1 (0.9509 - 0.0095X_1 - 0.0176X_2 - 0.0895X_4 + 0.0133X_4^2)$
3. $OD_3 = 1.400 + 0.1 (0.9509 - 0.0108X_1 - 0.0176X_2 - 0.0905X_4 + 0.0137X_4^2)$
4. $OD_4 = 1.400 + 0.1 (0.9509 - 0.0106X_1 - 0.0163X_2 - 0.0906X_4 + 0.0164X_4^2)$
5. $\frac{1}{3}(OD_2 + OD_3 + OD_4) = 1.400 + 0.1 (0.949 - 0.010X_1 - 0.017X_2 - 0.090X_4 + 0.014X_4^2)$
6. $OD_5 = 1.400 + 0.1 (0.9750 - 0.0068X_1 - 0.0097X_2 - 0.0890X_4 - 0.0046X_2^2 + 0.0048X_3^2 + 0.0106X_4^2 + 0.0050X_2X_3)$

Internal Diameter (For locations see Figure 5) - Figures 11 and 12

7. $ID_1 = 0.300 + 0.1 (0.7778 - 0.0028X_3 + 0.0583X_4 - 0.0060X_1X_4)$
8. $ID_2 = 0.300 + 0.1 (0.7841 + 0.0544X_4 + 0.0040X_4^2 - 0.0065X_3X_4)$

External Bond Strength (F = pounds force to pull 1/4 in. stud) - Figure 13

9. 1-1/4 in. from cap: $F = 693 + 111.4X_4$
10. Center of piece: $F = 913 - 34.4X_2 + 58.7X_4 + 53.7X_1X_4$
11. 1-1/4 in. from base: $F = 1011$

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Appendix B (Cont'd)

Internal Bond Strength (F = pounds force to pull a modified .19 in. stud)
Figure 14

- 12. 1-1/4 in. from cap: $F = 477$
- 13. Center of piece: $F = 674 + 64.9X_1 - 53.2X_4$
- 14. 1-1/4 in. from base: $F = 638 + 91.2X_1$

Aluminum-Nickel Diffusion Thickness (d)

- 15. Internal at center of piece. Figure 21
 $\ln 1000 (d+0.001) = 4.04 + 0.71X_1 + 1.08X_2 + 0.40X_4 - 0.66X_1X_4$
- 16. External at center of piece. Figure 22
 $\ln 1000 (d+0.001) = 2.70 + 0.72X_1 + 0.68X_2$

Internal Cladding Thickness at Center (c) Figure 23

- 17. $c = 30.90 + 2.69X_4$

External Cladding Thickness at Center (c) Figure 24

- 18. $c = 31.40 + 0.82X_2 - 3.27X_4$

Internal Closure Zone Cladding Thickness (c) Figure 25

- 19. Over Cap
 $c = 5.20 (8.562 - 0.607X_4)$
- 20. Over Core
 $c = 5.20 (5.999 - 0.620X_4)$

External Closure Zone Cladding Thickness (c) Figure 26

- 21. Over Cap
 $c = 5.20 (8.926 - 0.309X_4)$
- 22. Over Core
 $c = 5.20 (6.205 - 0.255X_1 - 0.775X_4)$

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