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FABRICATION OF HOT DIE SIZE DIFFUSION BONDED FUEL ELEMENTS FOR PRODUCTION TEST IP-546-A

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FABRICATION OF HOT DIE SIZE DIFFUSION BONDED FUEL ELEMENTS FOR PRODUCTION TEST IP-546-A

by

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INTRODUCTION

Hot die sizing (HDS) is a process being considered at Hanford to replace or supplement the existing AlSi brazing process. Hot die sizing consists of passing a preheated core-component fuel assembly through a cold die to bond the aluminum jacket to the core while passing a die plug through the internal bore to form the internal bond. Fuel end bonding is accomplished in a following step by applying heat and pressure to the sized fuel element. This report summarizes the fabrication of fuel elements for irradiation testing of hot die sized fuel elements as authorized by "Production Test IP-546-A, Irradiation of Hot Die Size Diffusion Bonded Fuel Elements," HW-75465.

SUMMARY

A total of twenty weighed and measured charges of hot die sized fuel elements were delivered to C-Reactor during July 1963. Three of the charges were thermocouple columns--two HDS charges and one AlSi brazed control charge. Seventeen columns consisted of matching alternating HDS and AlSi pieces in the downstream half of the charge. The upstream half of each of the 17 columns was made up of standard AlSi brazed fuel elements.

DISCUSSION

A total of 372 fuel elements was sized for irradiation testing of hot die sized fuel. Cores taken from alternate positions from the HDS core rod stock were assembled as AlSi brazed control elements for the HDS fuel. In order to maintain identity for matching finished pieces, all cores were identified prior to assembly and ingot, rod, and position numbers were recorded for each core. After assembly was complete, the finished pieces were paired in the downstream half of each of the 17 reactor charges. Standard AlSi fuel with modified end facing was used in the upstream half of each of the columns. Dimensions of modified C5N AlSi brazed control elements are shown in Figure 1. An additional three charges, two HDS and one AlSi, were prepared as thermocouple columns.

Hot die sized fuel element assembly detail will be discussed in the order in which the fuel was processed. AlSi Pilot Plant and Engineering personnel were used to fabricate the HDS and AlSi control pieces. Process data is recorded in Production Test Notebook HW-75465 B.

Hot die sizing fuel element assembly technique was modified to improve cap end bonding by the insertion of an aluminum-silicon (AlSi) wafer, 10 w/o Si, at the ends of the core. Recent testing indicated improved bonding would be obtained between the nickel plated ends and the AlSi wafer. The AlSi wafer offered another important improvement in that it seals striations opening on the core ends. It has also been observed that nickel plate over oxide filled striations has been broken due to the weak structural strength of the oxide and other impurities in the striation.

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Uranium Cores

Model CDB2N (HDS) and C5N cores were tested for transformation and grain size by the standard method of testing with the UT-2 tester. After testing, the pieces were pickled with nitric acid and inspected for striations and cracks to remove defective cores prior to plating.

Nickel Plating

Uranium cores were electro-nickel plated with a 0.8 mil nickel plate in a Watt's bath as described in HW-75465 D. After plating, the cores were inspected for cracks and other discontinuities in the plate.

Component Cleaning

Cap and can components were cleaned by caustic etching and deoxidizing to (1) prepare the inner surfaces for diffusion bonding to the nickel plated core, (2) to produce a matte finish on the can outside surfaces to increase the adherence of the Aqua-Dag lubricant coating, and (3) to change the cap and can dimensions to facilitate sizing. Cap and can cleaning technique used for this test is listed below:

1) Degrease - cold trichloroethylene
2) Caustic etch to reduce can outside diameter by 0.003 to 0.007 in. and cap outside diameter by 0.007 to 0.010 in. Etch solution: 4 to 5 oz. Aluminum per gallon at 50 to 60 C.
3) Flowing hot water rinse
4) Flowing cold water rinse
5) Dip in caustic etch solution (No. 2 above) 10 to 20 seconds
6) Flowing hot water rinse
7) Flowing cold water rinse
8) Deoxidize in Diversey 514 solution, 16 ± 3 oz per gal. for 5 to 15 min.
9) Flowing cold water rinse
10) Second flowing cold water rinse
11) Methanol rinse
12) Second methanol rinse
13) Hot air dry

AlSi wafers prepared from standard makeup AlSi were cleaned as follows:

1)	Caustic etch:	Turco 4181-19, 2 oz. per gal. at 65 to 70 C for 30 sec.
2)	Water rinse	
3)	Caustic etch:	Dip in No. 1 solution for 4 sec.
4)	Nitric-hydrofloric acid:	30% HNO_3, 0.5% HF, and 69.5% water for 1 min.
5)	Nitric acid:	20% for 2 min.
6)	Water rinse	
7)	Methanol rinse	
8)	Air dry	

[redacted]

Since the thin AlSi wafers had a tendency to stick together, baskets of wafers were agitated throughout the above cleaning cycle. After cleaning, all components were handled with clean cotton gloves.

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

After nickel plated core inspection and component cleaning, the fuel components were assembled. An AlSi wafer was inserted between the can base and the core and between the cap and the core. The assembly was then dipped in an Aqua-Dag solution (four parts water to one part Aqua-Dag) and allowed to air dry. Then the lubricated pieces were placed in the vacuum dryer and dried for a minimum of 30 minutes at a temperature of 100 to 125 F in a vacuum of 1 mm Hg. Just prior to sizing, the vacuum chamber was backfilled with argon to reduce component oxidation during fuel assembly preheat. Extra cores at shift end were assembled and held overnight under vacuum and then lubricated and sized the next morning.

Sizing

Immediately after vacuum drying and argon backfilling, the lubricated fuel assembly was fitted in the carrier, commonly called a heat shield. No more than ten assemblies were allowed to be out of the dryer chamber at a time to reduce the possibility of air drafts from drawing argon from the backfilled cans. Fuel assemblies were loaded in the preheat furnace at four minute intervals.

Concurrent with fuel assembly preparations and loading the first three assemblies in the preheat furnace, preparations to the sizing press were made. The external die (cold) was placed on the press platen, positioned with the lubricated (Fiske 604) alignment tool, and bolted down. The internal die was placed on the support stem and both dies were then lubricated with Fiske 604 extrusion lubricant making sure that the lubricant covered the approach angle and land. The external die was always lubricated with the lower ram at least 1/8 inch below the die to insure complete lubrication of the land. A heated die case was then placed on the die to raise its temperature to about 150 F. After the first piece was sized, no more heating was necessary to maintain die temperature.

During the last four minutes before sizing the first piece, the lower ram was cycled from three to six times to change the cold hydraulic oil in the lower cylinder. Cycling the ram increased the slightly heated oil temperature to about 125 F (warm to touch) by friction and warmed up the lower cylinder-ram assembly. Low temperature hydraulic oil was very viscous and could create enough back pressure on the lower ram to cause severe cap upsetting and failure to size the piece. An oil heater has since been installed to hold the bulk oil temperature about 100 F; however, it was necessary on later sizing runs to cycle the lower ram to heat the lower cylinder and piping prior to sizing.

Fuel assemblies were heated in the air resistance furnace set at 680 C for 12 minutes and then sized. After sizing, the piece was air cooled for 20 seconds with compressed air while in the down position. The cooled fuel element was then pushed back through the die and unloaded from the press. Cooling the piece reduced its over-all diameter and permitted pushing it through the die with a minimum of galling. The sized piece was then placed on the die ejector. After the inner die plug was removed, the hot die sized piece was set in the holding rack to air cool.

After the carrier parts and fuel elements were removed from the press, preparations were made to size the next piece. These preparations consisted of removing the upper ram tip, placing it on the platen to cool, and replacing it with a cool one. An inner die was fitted on the die support stem, the lower ram lowered just below the external die, and the inner and outer dies were lubricated. Since lubrication played a very important part in producing a good finish on the sized piece, several precautions were taken to maintain good appearance. Generous quantities of Fiske 604 were used so that enough lubricant was available for the entire fuel length. Sufficient quantity was evidenced by lubricant boiling in the well between the can and die during sizing. The lower ram tip was inspected periodically for upset metal on the edges. It had been observed that small amounts of upset metal on the lower ram removed lubricant from the external die land and produced galling on the piece in corresponding locations. Upset metal on the lower ram was removed with emery cloth.

Conditions for hot die sizing fuel elements for this production test material are itemized below.

TABLE 1

Summary of Hot Die Sizing Process Conditions and Limits

Preheat Furnace Temperature:	680 C ± 10 C
Preheat Time:	12 ± 0.5 min.
Ram Speed:	65 ± 3 in./min.
Wall Reduction:	45.5%
Component Lubricant:	Aqua Dag: 1 part to 4 parts water
Die Lubricant:	Fiske No. 604

Solvent Cleaning

Sized fuel elements are coated with considerable amounts of lubricant, both the dry and oil base, which are messy and contaminate anything they contact. Stoddard's solvent has been found to dissolve the lubricant and after a soak of about five minutes, the residual Fiske 604 and Aqua Dag can be wiped off. All the fuel elements were cleaned by washing with Stoddard's solvent prior to facing to prevent lubricant from being deposited in the facing lathe collets, and spreading to other cleaned fuel elements. The inner surface was cleaned by brushing with a test tube brush.

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Trim

Cleaned fuel elements were faced to remove the sizing flash and to prepare them to accept the end bonding die cups. Each sized piece has about three inches of internal and external flash at the top which was removed along with about 0.010 inch of the cap wafer itself. The base end was faced to remove upset metal to permit fitting the end bond die plugs and cups.

End Bonding

Sized fuel elements were end bonded as soon as possible after sizing, usually on swing shift immediately following day-shift sizing. In the event it was impossible to end bond, the pieces were then held in the vacuum dryer until ready for end bonding.

Preparations for end bonding consisted of (1) turning on the end bond press heaters about three hours before scheduled use, (2) numbering the pieces to insure maintaining individual piece identity, (3) marking the cap end to insure cap end identity and placing the pieces in the end bond press uniformly, and (4) fitting the pieces with differential thermal expansion die plugs and cups.

Pieces fitted with end dies were preheated in the air resistance furnace set at 680 C for 5-3/4 minutes and then transferred to the end bond press where they were heated and subjected to end loading to produce end bonding. Bonding on the lateral surfaces near the ends was produced by heat plus the pressure exerted by the difference in expansion between the fuel piece and the die cups and plugs. After applying a pressure of four tons per square inch on the end of the piece for 4-1/2 minutes, the piece was removed and water quenched. The die cups and plugs were then removed.

End bond process limits for producing the production test material are itemized in Table 2 below:

TABLE 2

Summary of End Bonding Process Conditions for HDS Fuel Elements

Preheat Furnace Temperature:	680 ± 10 C
Preheat Time:	5-3/4 ± 1/4 min.
Press Head Temperature:	655 ± 5 C*
Press Time:	4-1/2 min. ± 10 sec.
Pressure on End of Piece:	4 ± 0.25 $T/in.^2$**

* Difficult to hold (actual head temperature of 655 ± 10 C was maintained)
** Gage setting 464 ± 29 psig

Facing

End bonded pieces were faced to remove the slight back extrusion produced at the inner surface where the inner die plug and the outer die cup fit together. In addition to the 1/32 inch projection, about 0.010 inch of the cap and base were removed to clean up the ends. Each piece was then stamped with its core number.

Since there was a slight change in the weld technique originally intended, it was necessary to reface the base ends after nondestructive testing to provide weld bead relief for charging the pieces end-to-end. The base end contour produced is shown in Figure 2 giving over-all final fuel element dimensions.

Nondestructive Testing

Since cap thickness on HDS fuel elements is less than that of AlSi bonded fuel, it was necessary to test closure integrity (UT-4 test) prior to welding. Previous tests on welded pieces indicated considerable difficulty in detecting possible closure discontinuities due to the rounded surface of the weld bead. Nineteen pieces were rejected by the UT-4 tester for having more than 18 defective discs and/or 12 consecutive defective discs. Reject limits for AlSi brazed fuel elements are 22 defective discs and 15 successive bad discs. Seventeen AlSi controls were rejected for defects in the closure zone. There appeared to be good correlation between the UT-4 test and welding because UT-4 reject pieces were obvious weld rejects in most cases.

Hot die sized fuel elements were tested for end bond integrity and clad thickness on the prototypic UE-3 tester located in the 326 Building. The tester was set up to detect an unbonded area 1/4 inch in diameter. Ten pieces were rejected for defective end bonds--five cap end and five base end. Three pieces were rejected for thin internal cladding, less than .025 inch. Reduced clad thickness was later discovered to have been caused by marring the inner cladding during the time the die remover was being repaired and a steel rod was used to punch out the internal end bonding dies.

Hot die sized fuel elements and the AlSi brazed control elements were bond tested on the Pilot Plant bond tester. The HDS pieces were tested twice, once at a high sensitivity setting and again at the standard setting along with alternate groups of AlSi brazed control elements. Total bond count data for HDS and AlSi brazed control pieces tested using the standard setting are listed in Table 3 below. Any piece having a total count over 75 was rejected.

TABLE 3

Total Bond Count Data for HDS Fuel Elements

	HDS Fuel		AlSi Brazed Fuel	
	Internal	External	Internal	External
Number of pieces tested	272	272	783	783
Pieces having less than 30 counts	262	268	755	766
Pieces having 30 counts or more	10	4	28	17
Pieces having more than 75 counts	9	1	2	0
Pieces having more than 100 counts	9	0	2	0
Pieces having more than 200 counts	7	0	2	0

[redacted]

Welding

Fuel element welding methods were changed to improve closure integrity. Rather than use the step cut originally intended, the pieces were faced flat and weld bead relief was provided by contouring the base end as previously mentioned. The HDS pieces were welded with the Pilot Plant DC welder at conditions itemized below:

Amperage:	
External Weld:	140 amps, minimum
Internal Weld:	125 amps, minimum
Inert Gas Shielding:	95% Helium and 5% Argon
Turntable Speed:	20 ± 1 rpm
Passes:	2 passes plus crater fill

Weld inspection was delayed until after etching to aid in detecting discontinuities in the weld bead. Etching is described in the following section. As a result of etching, numerous small voids were noted in the welds. Since these voids resembled "pinholes", inspection was difficult and all pieces having voids were set aside. The pits were caused by etching of localized impurities. Examination of the minute pits and voids revealed that they were only on the surface, but because of weld inspection difficulty, the pieces were rewelded and inspected again.

Cleaning and Etching

The residual film on hot die sized fuel elements resisted cleaning by conventional methods. Prior to learning that Stoddard's solvent removed extrusion lubricants, a nitric-hydroflouric acid mixture was used which has always been difficult to handle. Stoddard's solvent does not remove all traces of the lubricants, making it necessary to use a mild etchant prior to attaching self-support projections. The use of a nitric acid, caustic, nitric acid sequence has cleaned HDS fuel elements very well. Details for cleaning are itemized in Table 4 below:

TABLE 4

Hot Die Sized Fuel Element Cleaning and Etching Process

1. Nitric Acid

Concentration:	55 - 60%
Temperature	75 - 85 C
Time	5 ± 1/4 min.

2. Water Rinse

3. Caustic Etch

Concentration:	2.5 - 3.0 oz. Turco 4181-19/gal.
Temperature:	80 - 85 C
Time:	Sufficient to remove 0.5 - 1.5 mils from surface (about 2-1/2 min.)

4. Water Rinse
5. Nitric Acid

Concentration:	15 - 30%
Temperature:	80 - 85 C
Time:	12 - 15 min.

6. Water Rinse
7. Dry

Self-Support Projection Attachment

Self-support projections were attached to both the HDS and AlSi control pieces according to the standard procedure for model C5N fuel elements with one exception. The finished length for both models was slightly longer than that of the standard fuel element. For this reason, the self-support projection rows were moved down from the cap 1/8 inch. Standard C5N self-support projections were attached 1-3/8 ± 1/16 inch and 5-3/8 ± 1/16 inch from the cap ends of both models.

Autoclave

Hot die sized fuel elements were autoclaved a minimum of 56 hours. The autoclave cycle was increased to further assure fuel element jacket and closure integrity.

Metallography

Twenty HDS fuel elements were sectioned and polished for bond examination. Four sections were taken from each sample:

- Section A - Longitudinal cut through diameter of 1/2 inch base end section.
- Section B - Longitudinal cut along the radius of one-inch long section taken next to Section A above.
- Section C - Same as Section B but taken from cap end.
- Section D - Same as Section A but taken from cap end.

End sections were examined for the following reasons:

1. To determine the quality of bonding on end surfaces.
2. To examine closure integrity and estimate degree of AlSi flow between cap and inner and outer cladding.

3. To examine lateral surface bonding in the regions under the differential thermal expansion dies and in the area adjacent to the dies.
4. To determine the degree of AlSi wafer bonding to the nickel plated core and to the aluminum end cladding.
5. On previous examinations, bonding of the six-inch center section was similar to the "B" and "D" Sections.

Bonding of the AlSi wafer (10 w/o Si) to the aluminum cap and base and to the nickel plated core was of particular interest. The AlSi wafer was completely diffusion bonded in all samples of fuel elements processed according to previously mentioned sizing and end bonding conditions. In spite of the fact that the AlSi wafer was not heated to its melting point (~ 557 C), AlSi-nickel diffusion occurred. As had been previously observed, AlSi and nickel appear to diffuse more readily than aluminum and nickel.

The aluminum-AlSi junction was examined to determine degree of bonding. On fuel elements heated sufficiently to melt the AlSi wafer, wetting of the aluminum occurred. Wetting was distinguished very readily and resembled standard dip brazing wetting. There was some evidence of diffusion between the aluminum alloy 8001 and non-wetted AlSi, but could not be determined readily by optical microscopy. Good mechanical contact was observed in all cores.

On the cap end, the AlSi wafer was forced up and in between the cap and the fuel lateral surface cladding. The average height the AlSi was folded up between the cap and the outer and inner cladding was 0.035 inch and 0.041 inch, respectively. There was always more wetting on the can and spire tube sides of the folded AlSi wafer than the cap side. This increased wetting was believed due to the higher temperature of the clad side and to more friction between the inner and outer tubing and the AlSi than between the cap and the AlSi wafer. The estimated amounts of wetting in inches on both sides of the folded up AlSi wafer are listed below:

		Cap Side	Can or Spire Side
External -	Range	0-0.040 in.	0-0.040 in.
	Median	0.004 in.	0.008 in.
Internal -	Range	0-0.050 in.	0-0.060 in.
	Median	0 in.	0.004 in.

Base end bonding, Section "A", was very much more consistent than cap end bonding. With the exception of one fuel element, the AlSi wafer was completely bonded to both the nickel plated core and the can base. Better and easier bonding is produced because there is less aluminum and nickel oxide to hinder diffusion than on the cap end.

Examination of the one-inch section taken from below the cap and base, Sections "D" and "B", respectively, revealed good nickel-uranium and aluminum-nickel diffusion. No large-scale cracking was observed either in the end bond zone under the differential thermal expansion dies or in the area adjacent to the dies. Cracking has been observed in aluminum-nickel diffusion when the layers become quite thick because of over heating and when shear stresses have been applied to fuel elements because of press misalignment.

Preparation of Test Charges and Fuel Element Dimensions

Twenty weighed and measured charges for testing hot die sized fuel elements in-reactor were prepared. Three of the charges were thermocouple columns--two HDS charges and one AlSi brazed control charge. Seventeen charges consisted of matched alternating HDS and AlSi pieces in the downstream half of the charge. AlSi brazed pieces were used in the upstream half of the charges.

Weights and measurements of the twenty columns were recorded and submitted to Applied Mathematics, Hanford Laboratories, for analysis. Average measurements ($\bar{x}$) and standard deviations (S) for the HDS and AlSi controls are listed in Table 5 below.

TABLE 5

Pre-Irradiation Measurements of Hot Die Sized and AlSi Control Fuel Elements

	HDS		AlSi	
	$\bar{x}$	S	$\bar{x}$	S
Weight (grams)	3980.7	4.857	3680.3	3.433
Length (in.)	9.0463	0.0082	8.9302	0.0107
OD_1*-Max. (in.)	1.4932	0.0010	1.4928	0.0012
OD_1-Min.	1.4914	0.00094	1.4894	0.00097
OD_1-Difference	0.0018	0.0011	0.0034	0.0016
OD_2*-Max.	1.4913	0.00098	1.4928	0.0012
OD_2-Min.	1.4897	0.00099	1.4889	0.0012
OD_2-Difference	0.0016	0.0013	0.0039	0.0019
OD_3*-Max.	1.4925	0.00099	1.4941	0.0011
OD_3-Min.	1.4912	0.0011	1.4906	0.0010
OD_3-Difference	0.0013	0.0009	0.0036	0.0015
Warp	0.0020	0.00116	0.0041	0.00173
ID-5*	0.3783	0.00071	0.3799	0.00042
ID-6*	0.3788	0.00063	0.3802	0.00048
ID-7*	0.3793	0.00071	0.3804	0.00055

*OD-1 and ID-5: 1 in. from base end
OD-2 and ID-6: center of piece
OD-3 and ID-7: 1 in. from cap end

[redacted]

Bond Strength

External bond strength measurements were taken on 34 HDS fuel elements. The average bond strength for the pieces examined was 788.3 pounds force to pull a 0.190 inch diameter stud corresponding to a tensile strength of 26,800 psi. Average bond strength for C5N control elements was 447.2, 15,205 psi tensile strength. Bond strength data are summarized as follows:

	HDS Fuel	AlSi Control
Number of Pieces Tested	34	5
Average Bond Strength		
Expressed as Pounds Force to Pull a 0.190 in. Diameter Stud	788.3	447.2 lb
Equivalent Tensile Strength (psi)	26,802	15,205 psi
Standard Deviation (lb)	165	126.5
Minimum Bond Strength Expected		
Pounds Force (lb)	293	67.7
Equivalent Tensile Strength (psi)	9,962	2,302
Minimum Observed Bond Strength		
Pounds Force	420	180
Equivalent Tensile Strength (psi)	14,280	6,120

C. A. Strand

Engineer
Process Development Unit
Fuels Engineering Operation

CA Strand:skc

FIGURE 1

MODIFIED C[illegible] AlSi [illegible] FUEL ELEMENT

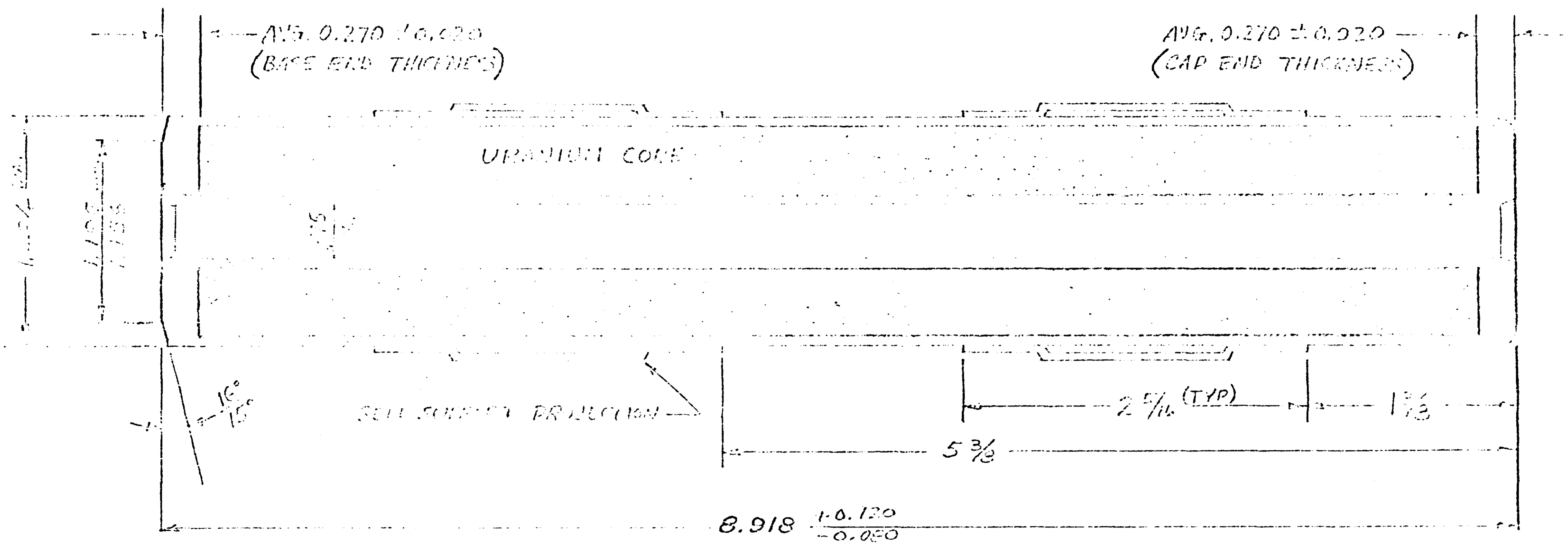
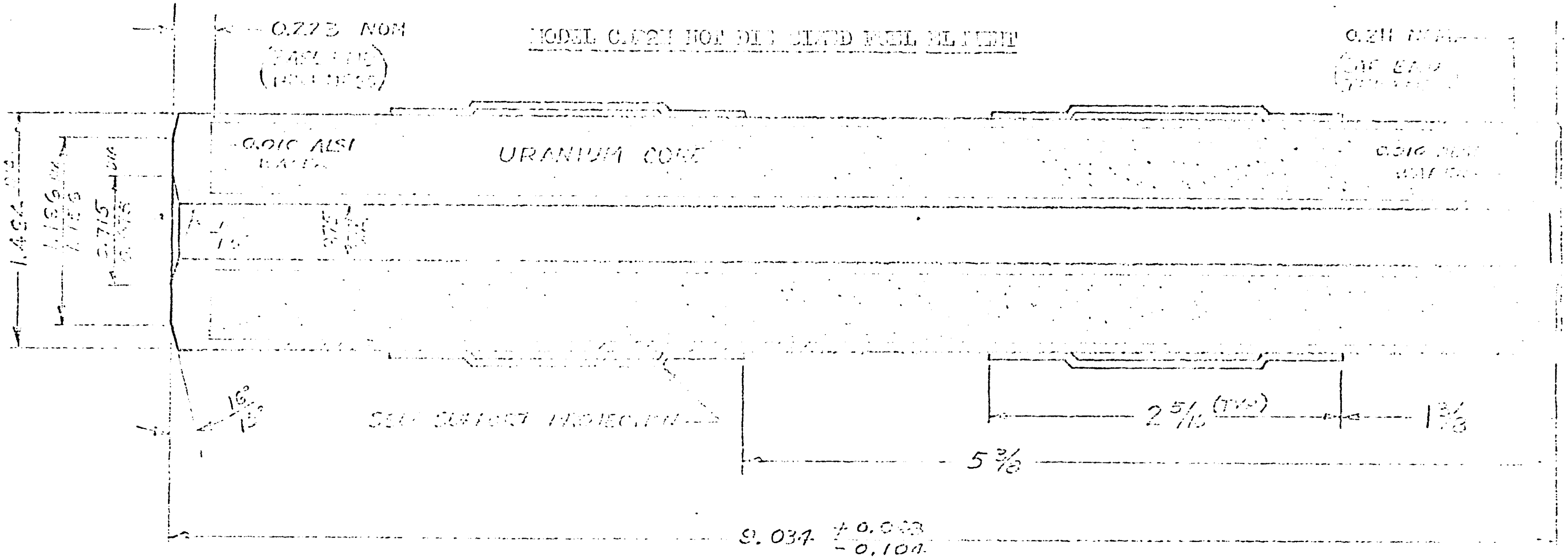


FIGURE 2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