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JANUARY, 1963

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STATUS OF ALLOYED DINGOT PROGRAM
JANUARY 1963

by

E. A. Weakley

Product Engineering Operation
Fuels Engineering Operation
Production Fuels Section
IRRADIATION PROCESSING DEPARTMENT

January 11, 1963

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DECLASSIFIEDINTRODUCTION

This report summarizes and highlights the more important milestones, development programs, performance characteristics, and properties of dingot uranium and its use in the Hanford reactors from 1955 to the present. For the benefit of those unfamiliar with the terms ingot and dingot uranium as used in this report, ingot uranium refers to the metal made at the Fernald Plant of the National Lead Company by remelting under vacuum a mixed charge of solid scrap, briquetted scrap, and derby metal (the product of the UF_4 bomb reduction), while dingot metal refers to the metal made by Mallinckrodt Chemical Works by reduction of UF_4 to metal which is used directly (after suitable forming into convenient size for rolling) without the intermediate vacuum remelting step.

Initial reactor testing of low hydrogen (<2.5 ppm) dingot solid cores^{1,2,3,4} and I & E cores⁵ between 1955 and 1958 showed that dingot cores were less dimensionally stable than ingot cores^{6,7,8,9} with respect to warp, diameter change, and bumping. Diameter increases and particularly surface bumping observed on the dingot fuel elements were believed related to the larger grain size.⁸ The approximate 18" diameter x 18" high dingots used in the initial production tests were produced in the old Mallinckrodt Chemical Works (MCW) St. Louis Destrehan Street Pilot Plant and were forged to a billet size suitable for rolling in the NLO rolling mill. When the new MCW Weldon Spring Production Plant started up in 1958, MCW was unable to duplicate the production of low hydrogen dingots and it was necessary to terminate the reactor charging⁵ due to lack of low hydrogen cores.

During the remainder of 1958 and most of 1959, MCW developed methods at the Weldon Spring Plant for (1) fabricating low hydrogen (<2.5 ppm) dingot by lengthening the bomb preheating time and by vacuum outgassing core blanks, (2) refining the beta heat treated grain size by the addition of about 150 ppm iron and 100 ppm silicon to the reduction bomb, and (3) gamma phase extrusion rather than forging of dingots into billets for rolling at Fernald.^{10, 11} In the fall of 1959, the first production test quantities of low hydrogen (by long bomb preheat) alloyed or additive* dingot OIIN cores were available for shipment to HAP0.

The initial production test¹² for alloyed dingot cores authorized the charging of 12 monitor tubes of alternated alloyed dingot and ingot control elements. These were charged in January 1960. A total of 50 tons of lot-charged dingot material was charged starting in April 1960. Post-irradiation measurements of the alternate dingot-ingot monitor columns indicated that under normal exposure conditions the ingot and alloyed dingot fuel elements exhibited similar dimensional distortion characteristics.¹³

*Throughout this report the use of the words alloyed or additive dingot refers to the material made by adding small quantities of iron (~ 150 ppm) and silicon (~ 100 ppm) to the metal.

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Based on the favorable results of four tubes discharged at low exposure (361-369 MWD/T) from the initial alloyed dingot production test, a larger scale test¹⁴ for lot charging 360 tons (a maximum of 60 tons per month) in D and DR Reactors began in May 1960.¹⁵ In July, 1960, the test was revised to permit lot charging of alloyed dingot in B Reactor¹⁶ and in October, 1960, an additional 540 tons were authorized for charging in all the old reactors at a maximum rate of 90 tons per month.¹⁷

The rupture evaluation of lot charged alloyed dingot fuel elements was based on a "sequential" test design to establish whether or not dingot was equal in quality or two fold worse than ingot. Dimensional characteristics of alloyed dingot and ingot elements after irradiation were compared using data from the Quality Certification Program.

By mid May, 1961, after irradiation of 510 tons of dingot to goal exposure, eight failures had occurred in both the alloyed dingot and ingot control material. Since, at this point the performance of the dingot and ingot controls was essentially equivalent, the program was modified¹⁸ to (1) continue sequential testing of alloyed dingot, (2) use dingot in all core models, and (3) authorize unlimited quantities of dingot uranium following the completion of the 90 ton per month test with the provision that no more than two-thirds of any one core model be dingot. However, the provision to authorize unlimited quantities of dingot uranium was modified¹⁹ for administrative reasons to limit the tonnage of alloyed dingot uranium produced during FY-1962 to 125 tons/mo. Also at this time, the charging of dingot uranium in the K reactors was authorized.

By October 1961, it became apparent that the over-all performance of dingot uranium was deteriorating²⁰ and that, what was more important, a type of failure was occurring in dingot uranium which had not been previously encountered in recent production irradiation. At this point K reactor charging of dingot was discontinued.

By the end of May, 1962, approximately 1310 tons of "O" size alloyed dingot cores and 173 tons of "K" size dingot cores had been discharged at goal exposure and 198 tons of "O" size and 123 tons of "K" size dingot cores were still being irradiated. Based on the established sequential analysis²¹ and after 21 dingot and 10 ingot control failures in nonbumpered fuel elements, the dingot evaluation was completed with the conclusion that dingot fuel performance was a factor of about three worse than ingot.²² In addition, four dingot failures and three ingot control failures had occurred in bumper fuel elements. Not only was the rupture performance of dingot uranium approximately three times worse than ingot uranium but also the problem was aggravated by an outbreak of six low exposure failures (<37% of goal) in a four week period in April and May, 1962, and by the occurrence of six unusual transverse split type failures which had not been observed in ingot metal. Thus, in May 1962, the charging of dingot uranium was discontinued to evaluate the problem from a technical viewpoint and to re-evaluate the incentives for proceeding with the use of dingot uranium in the Hanford reactor.²³ In October, 1962, the decision was made to discontinue the use of dingot uranium at Hanford because it did not appear likely from a technical point of view that dingot could be farther improved to make it perform better than ingot uranium, and because economic incentives were lacking.²⁴

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As of January, 1963 there was on hand at HAPU approximately 105 tons of canned dingot uranium fuel elements and 270 tons of bare dingot cores. Plans are under-way to subject this material to stringent testing standards and, in the case of the bare cores, to a more optimum canning cycle for dingot; and to reactor charge the material during the calendar year rather than scrap it. Also, approximately 940 tons of dingot uranium in various stages of fabrication are at NLO and MCW.⁴⁰ It is planned to utilize a portion of this material.

SUMMARY

The first alloyed dingot uranium was shipped to HAPU in September 1959, canned in December 1959 and reactor charged in January 1960. The authorized shipments of alloyed dingot uranium were increased in steps from 25 ton/month in January 1960, to 60 tons/month in April 1960 and to 90 tons/month in October 1960. In June 1961, dingot production was restricted to 125 tons/month through FY 1962.

The total tonnage of alloyed dingot discharged and monthly status of cores being irradiated is shown in Figure 20.

As of June 1, 1962, 22 nonbumpered and 4 bumpered failures had occurred in alloyed dingot elements and 11 nonbumpered and 3 bumpered failures in the ingot control elements as shown in Table I. The fuel element performance level, q , of non-bumpered dingot elements was 2.6 times worse than that of the ingot control elements at that time.

To compare ingot and alloyed dingot elements as to failure performance a sequential test was conducted to show either that (1) dingot was equivalent to normal ingot elements with 90% confidence or (2) dingot was two fold worse than normal ingot with 95% confidence. In April 1962, after 20 nonbumpered dingot failures and 10 nonbumpered ingot failures, the sequential test fell below the lower control limit, where dingot was considered two fold worse than ingot metal.

Six of the 26 alloyed dingot failures were core split failures (primarily in the transverse direction) while none of the control ingot failures failed by core splitting. From examinations it is believed that transverse split failures occur when water reaction in shrinkage cracks (or hydrides) wedges the metal apart from the inside surface outwards to the exterior surface. During the quench after salt bath heat treatment, beta-alpha transformation results in a volume decrease that produces small randomly oriented shrinkage cracks in the center portion of the solid dingot core blanks. In normal ingot uranium this volume change occurs primarily at inclusion stringers (mainly carbides) which are oriented in the longitudinal direction near the center of the solid core blank. The majority of the shrinkage cracks are removed during the core drilling operation, but some of the randomly oriented cracks remain on the I. D. surface of dingot cores. Uranium hydrides are also found near the I. D. surface on some of the dingot cores that may act as stress risers.

Alloyed dingot uranium is known to etch at a slower rate than ingot metal and the attack by molten metals (lead and Al-Si) in the canning baths may also produce a poor bond, especially on the I. D. surface, that is susceptible to water entry to the core. This slower etch rate of dingot is due to the high purity (low carbon content) of the metal.

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Table I. COMPARISON OF DINGOT AND INGOT FAILURES
JUNE 1, 1962

	<u>Nonbumpered Elements</u>	
	<u>Alloyed Dingot</u>	<u>Ingot Control</u>
<u>Failures</u>		
Side Hot Spot and Side Other	5	9
Internal and End	8	0
Core Split	5	0
Unclassified and Unknown	<u>4</u>	<u>2</u>
	22	11
<u>Severity (ARI)</u>	107	137
<u>q (Failures/ARI)</u>	0.206	0.080

	<u>Bumpered Fuel Elements</u>	
	<u>Alloyed Dingot</u>	<u>Ingot Control</u>
<u>Failures</u>		
Internal and End	2	3
Core Split	1	0
Unclassified and Unknown	<u>1</u>	<u>0</u>
	4	3
<u>Severity (ARI)</u>	58	76

q

Insufficient data to draw any conclusions regarding fuel element performance.

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Analysis of Quality Certification and Production Test post-irradiation data has shown that (1) there is very little difference in warp between ingot and dingot, (2) the average OD_2 change for dingot cores shipped in 1960 was higher than ingot cores, and (3) the tube filling capacity of dingot was greater for dingot cores shipped in 1960 than for ingot cores. These data are shown in Figure 21.

The poorer rupture performance of dingot, the prevalence of low exposure failures, and the occurrence of six unusual transverse split type failures caused a cessation of dingot charging in the reactor in May 1962, and an intensive program of technical evaluation of the factors causing the poor performance was undertaken. The quality problems were considered to be closely associated with 1) quench cracks on the I. D. surface in dingot metal cores, 2) a lower chemical reactivity in dingot uranium, and 3) the presence of more brittle USi_3 inter-metallic compound in the bonding layers of dingot fuels as compared to ingot fuel. Process changes, modifications, and improvements which alleviate each of these undesirable conditions have been evolved and it is believed that their use would result in the production of a dingot metal fuel element that more nearly approaches the ingot fuel element in reactor performance. However, as of January 1963, no irradiation data on dingot fuel elements produced by the improved processes has been accumulated.

The process modification and improvements which are believed to affect the dingot uranium and result in a fuel element with better reactor performance characteristics are:

1. Elimination of Quench Cracks on the I. D. surface.

Quench cracks can be essentially eliminated in dingot cores by heat treating the core in hollow blank form and quenching into Houghto-K quenching oil at a temperature of 130 F.

2. Improvement of Chemical and Surface Reactivity

The surface reactivity of dingot uranium can be made to approach or equal that of ingot uranium by giving the fuel cores a post beta heat treatment anneal. This treatment re-precipitates inter-metallic compounds (probably U_6Fe and U_3Si), and increases the reactivity. Process conditions to accomplish this anneal include holding the cores under vacuum at 950-1000F for 8 hours.

3. Improved AlSi Bonding

In the AlSi canning process, dingot uranium fuel when canned by the same process as ingot uranium contains in the bond layers a greater amount of the more brittle USi_3 intermetallic compound than ingot fuel elements particularly on the inner surfaces of the bore. It is believed that this more brittle layer may have contributed to the poorer performance in the reactors. The condition can be alleviated by reducing the silicon content of the duplex furnace AlSi from 9.0 to 7.5 per cent and by increasing the duplex furnace lead temperature from 616 to 625C.

DISCUSSION

The dingot uranium process was developed in 1955 to produce high-purity metal by eliminating the vacuum-remelt-casting operation used in the ingot process. The cores produced by the early dingot process had two major drawbacks: a high hydrogen content and a large grain size. High hydrogen content cores increase the porosity levels of canned fuel element braze layers resulting in increased bond rejects and reduce the rupture resistant quality of the fuel elements by increasing the probability of water reaching the core in the event of a weld or jacket failure. Cores with large grain size are known to cause surface bumping and are believed related to diameter increases during irradiation.⁸ The hydrogen level of dingot uranium was controlled by a long bomb preheat (about 30 hours) in 1959, by an internal Mg wire used as an electrical resistance bomb preheater (12 to 15 hours), and/or by vacuum outgassing core blanks before beta heat treatment in 1960. The grain size was controlled in 1959 by the addition of about 150 ppm iron and 100 ppm silicon to the bomb charge. A flowsheet of the current ingot and dingot uranium process is shown in Figure 1.

A comparison of ingot and alloyed dingot uranium as to measurable preirradiation, canning, and irradiation characteristics follows:

I. Measurable Preirradiation Core Characteristics

A. Chemistry and Density

1. Hydrogen

Hydrogen values of normal ingot are usually less than 2.0 ppm due to the extraction of hydrogen from the metal during the vacuum-remelt-casting operation. Even though the average total hydrogen for normal ingot cores is 1.5 ppm, HAPC has analyzed ingot cores exceeding the desired maximum limit of 3.0 ppm as shown in Figure 6.

In 1959 and early 1960, MCW used a 30 hour bomb preheat to produce alloyed dingot metal with less than 2.5 ppm hydrogen. With previous bomb preheat cycles of about 8 hours, the hydrogen level of non-alloyed dingot averaged about 4 ppm. In May 1960, MCW began using an electrical resistance magnesium wire to internally preheat the bomb and were able to reduce the preheat times to 12 to 15 hours. The magnesium wire contains a small amount of aluminum and adds about 15 ppm of aluminum to the dingot metal. Dingots that analyzed greater than 2.4 ppm hydrogen were used in the National Lead Company of Ohio (NLO) ingot process for remelt or, after April 1960, vacuum outgassed as solid core blanks before beta heat treatment. Vacuum outgassing at approximately 0.15 mm

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of Hg and 950 F for greater than 16 hours reduced the average hydrogen level to less than 1.0 ppm and did not affect the grain size or grain orientation of the finished cores. Approximately 60% of the alloyed dingot cores received in 1961 and 1962 were vacuum outgassed.

Hydrogen is present in alloyed dingot metal as dissolved atomic hydrogen and as hydride, UH_3 . The apparent diffusivity of hydrogen during vacuum outgassing is lowered due to the disassociation of UH_3 ²⁴ and it becomes difficult to reduce the hydrogen content in the center portion of a solid core blank to below 3 ppm. As a result, some vacuum outgassed cores received at HAPC that have a total hydrogen value less than 2 ppm still have UH_3 stringers near the core I.D. as shown in Figure 16.

Core blanks absorb up to 0.5 ppm total hydrogen from the molten chloride salt bath.²⁴ The average total hydrogen content for non-outgassed dingot cores is 2.1 ppm and for vacuum outgassed dingot cores is 1.0 ppm. The hydrogen absorbed by the core blanks during heat treatment is concentrated near the surface resulting in finished cores having higher than average hydrogen concentration at the O.D. surface where it is readily available for contaminating the braze layer during canning.

About 37,000 additive dingot OIIIIN cores were prepared by MCW in mid May, 1961, by vacuum outgassing hollow core blanks and finished cores after beta heat treatment. Vacuum outgassing in hollow form after beta heat treatment appears advantageous since:

- a. it requires a shorter outgassing time,
- b. the hydrogen absorbed during the salt-bath treatment is removed leaving essentially a zero hydrogen concentration on the core surface.
- c. the UH_3 stringers found near the I.D. surface on some of the solid-blank-vacuum-outgassed cores are removed,
- d. the outgassing temperature is in the stress anneal range and should anneal out the residual quench stresses.
- e. the outgassing temperatures is in the precipitation anneal range which will precipitate intermetallic compounds, resulting in a change in surface chemistry that produces a faster nitric acid etch rate.

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MCW found that the dimensions of hollow blanks or cores changed after a vacuum-outgassing-after-beta-heat-treatment by the following amounts, suggesting that stress anneal had taken place:²⁵

<u>Dimension</u>	<u>Average Change</u>
Warp, T.I.R.	-1.4 mil
O. D.	+0.4 mil
I. D.	+0.9 mil
Length	-2.6 mil

Approximately 36,000 of the vacuum-outgassed-after-beta-heat-treatment OIIN cores were canned during the summer of 1961. As of the end of November, 1962, 97 percent of these fuel elements had been discharged at goal exposure without incident. Six Quality Certification columns of vacuum-outgassed-after-beta-heat-treatment cores were measured for dimensional changes during irradiation and no significant differences were found between the test cores and regular dingot cores.

2. Carbon, Iron, Silicon, and Other Impurities

Carbon, which is picked up during vacuum remelt and by scrap additions, is the major contaminant of normal ingot uranium. Dingot uranium averages less than 30 ppm carbon whereas normal ingot uranium averages between 400 and 500 ppm carbon, as shown in Figure 2. The maximum acceptable limit is 750 ppm carbon. Due to the inherently low carbon content of dingots only every 50th dingot has been analyzed for carbon since July 1960. Carbon occurs in uranium as uranium carbide inclusions, as shown in Figure 3. During rolling the carbide inclusions are strung out by plastic deformation into longitudinal stringers or striations.

The iron and silicon content of alloyed dingot uranium is understandably higher than in normal ingot due to the iron and silicon additions for grain refining. MCW attempts to control the alloyed dingot iron content to 150 ppm and the silicon content to 100 ppm. The maximum acceptable iron and silicon content is 185 and 140 ppm, respectively. Figures 4 and 5 show the average iron and silicon levels of alloyed dingot and normal ingot uranium. There is an upward trend in iron and silicon content of normal ingot uranium due to increased use of additive dingot metal as remelt stock in the ingot process.

Also included in Figures 4 and 5, are the average nickel and nitrogen levels of alloyed dingot and normal ingot metal. The maximum acceptable value for both nickel and nitrogen is

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100 ppm. The only trends noted were a slight decrease in average nitrogen content in normal ingot and a slight increase in average nitrogen content in alloyed dingot uranium.

The average values of other reported impurities from July 1959 through December 1960 are quite low as follows:²⁸

<u>Element</u>	<u>Average Impurity Level</u>		<u>Maximum Acceptable Limit</u>
	<u>Alloyed Dingot</u>	<u>Normal Ingot</u>	
Aluminum	15 ppm	-	150
Chromium	5	10	65
Copper	4	-	75
Lead	<3	-	--
Magnesium	<5	4	25
Manganese	5	10	25

3. Density

The average density of alloyed dingot uranium (19.00) is higher than that of normal ingot uranium (18.93) primarily due to the difference in carbon content.

B. Reactivity

Due to the higher density of dingot metal, the average reactivity, as measured by the 305 Test Reactor, of additive dingot cores was higher than that of normal ingot cores until August, 1961, as shown in Figure 7. The increase in reactivity of normal ingot cores is attributable to a slight contamination of the normal ingot stream with enriched uranium. The slightly higher reactivity of dingot metal prior to August 1961 is insufficient to raise the core temperature enough to cause dingot metal to be more rupture prone than ingot metal.

C. X-Ray Diffraction Data

One core from each incoming uranium lot is taken as an audit x-ray diffraction sample at HAPC and the following ten planes are measured for I-maximum in the longitudinal direction: 020, 110, 021, 002, 111, 112, 131, 023, 200 and 113. From January, 1962, on an additional eight planes were measured for I-maximum in the longitudinal direction: 130, 132, 133, 114, 150, 223, 152 and 312. Growth index values are calculated using the area-weight method (G_3). The average monthly growth indices (G_3) and texture coefficients of the 200 and 020 planes for normal ingot and alloyed dingot cores are shown in Figure 8, 9, and 10. The texture

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coefficient values of these two planes were plotted since they usually indicate any change in the normal beta heat treated structure. Uranium cores with a predominant 200-texture tend to shrink in length and cores with a predominant 020 texture increase in length. Cores having a strong positive G_3 value have a tendency to grow in length and cores with a strong negative G_3 value to shrink in length.

There is very little difference between the growth indices (G_2) for normal ingot and alloyed dingot cores as shown in Figure 8. Of interest, is the sudden rise in G_3 in January and February 1961 for both ingot and dingot that was due to a sudden rise in the 020 texture (see Figure 10) without a change in the 200 texture coefficient (see Figure 9). This rise in the 020 texture coefficient is believed due to some as yet unexplained change in the rolling mill at Fernald, since the rolling and rod straightening steps are the only identical processing operations in the fabrication of normal ingot and dingot cores.

Although the 200 texture coefficients for additive dingot are higher than for normal ingot cores the 020 texture coefficients in 1961 were also higher, which tends to counteract the effect of a higher 200 texture.

From reviewing the G_3 and the 200 and 020 texture coefficient data, it would appear that both normal ingot and dingot cores shipped in 1961 should have a greater tendency to grow in length than cores shipped to HAPO in 1960.

D. Grain Size

The average grain size of alloyed dingot uranium is consistently larger than the average grain size of normal ingot cores (see Figure 11) as measured by the FEDC Visual Grain Standards. However, since mid 1961 the UT-2 reject rate for alloyed dingot uranium compares quite closely with that of normal ingot cores (except for November 1961 and April 1962) as shown in Figure 12. When HAPO began UT-2 testing alloyed dingot cores shipped during a period when the quench conditions at the feed sites were not so well controlled (October to December 1960), the UT-2 reject rate jumped sharply to greater than 16% and then rapidly decreased to less than 2%. A portion of the rejects during this high reject period were due to malfunctions of the equipment, which were corrected by July 1961 with improved circuitry.

E. Mechanical Properties

MCW has recently completed impact tests on normal ingot, unalloyed dingot and alloyed dingot uranium and has shown essentially no difference between the types of uranium.²⁷ At temperatures below 200°F all of the samples tested exhibited brittle fracture.

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Impact tests on alloyed dingot uranium indicated that oil-quenching after salt-bath beta heat treatment produces uranium that is slightly more ductile than brine-quenched metal.²⁷

Tensile tests were also performed by MCW on dingot metal which contained varying amounts of iron and silicon and on normal ingot uranium. The effects of these various alloy contents on elongation and reduction in area are shown in Figures 13 and 14.²⁷ All of the dingot samples used were machined from standard beta heat treated core blanks that had been vacuum outgassed before heat treatment. As can be seen, as the alloy content increased the ductility decreased. It is possible that if the iron content exceeded 200 ppm, the uranium core would be in a brittle condition and susceptible to failure during thermal cycling. The ingot samples were machined from standard beta heat treated core blanks and even though the iron and silicon contents are 112 ppm and 73 ppm, respectively, the ductility is higher than for low iron and silicon dingot metal above 200F.

F. Cracks and Surface Inspection

1. Shrinkage Cracks

During the 130F brine quench after heat treatment, beta-alpha transformation results in a volume decrease that produces small randomly oriented shrinkage cracks in the center of solid alloyed dingot core blanks as shown in Figure 15. In normal ingot uranium this volume change occurs in solid core blanks primarily at inclusion stringers (mainly carbides) which are oriented in the longitudinal direction near the center of the solid core blank as shown in Figure 15. The majority of the shrinkage cracks are removed during the core drilling operation, but some of the randomly oriented cracks remain on the I. D. surface of alloyed dingot cores as shown in Figure 16. These shrinkage cracks can be avoided by hollow core blank heat treatment, 43 second air delay, and followed by quench into 130F Houghto-K oil or into 130 or 175F brine.²⁸

2. O. D. Surface Quench Cracks

In 1960, three incidents of O. D. surface cracked ingot cores (commonly called "mud-flat" cracks) were found by surface inspection at HAPO. These cracked ingot cores, as shown in Figure 17, were found to be caused by high iron contamination (500 to 630 ppm) and the cracks were up to 0.248 inch deep. Hypereutectoid concentrations of iron are known to produce severe cracking in uranium during quenching from the beta phase due to the formation of the brittle U_6Fe phase.²⁸ Failures have occurred in four out of the eight virgin ingot lots known to have contained surface quench cracks, but none of the failures could be specifically attributed to the surface cracks.

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In mid 1961, six dingot cores were found that had large random surface cracks up to 1/4 inch deep on a portion of the core surface. These cracks, as shown in Figure 17, were not as extensive as the surface quench cracks observed on ingot cores and the iron content was less than 185 ppm. It is believed that these cracks were due to corrosive attack of the core blanks during damp storage prior to heat treatment which produced cracks during heat treatment.²⁹ None of these surface cracks were associated with internal shrinkage cracks.

A partially developed circuit to detect surface quench cracks was installed on the UT-2 testers in January 1960. In mid April 1961, this test circuit was removed for further development work. "Mud-flat" testers suitable for production use were installed on all UT-2 testers in September 1961.

3. Surface Inspection

Visual inspection reject rates at HAPO for normal ingot and alloyed dingot virgin cores have been essentially comparable (averaging less than 0.56%) since February 1961 as shown in Figure 18. Prior to that date, the normal virgin ingot surface reject rates were about one percent higher than for alloyed dingot virgin cores due primarily to carbide inclusion stringers on the core surface (striations).

The surface inspection monthly reject rate at the feed sites has been better for alloyed dingot cores, which averaged less than 5 percent, than for the normal ingot cores, which averaged about 10 per cent prior to December, 1960, and less than 5 percent from December, 1960, to date.

II Canning Characteristics

A. Core Etch Rate

In April 1962, it was determined that the alloyed dingot core etch rate in nitric acid was significantly slower than for ingot cores. For example, for ingot and alloyed dingot OIIN cores (in separate baskets) etched in a vertical position in 57.5 wt. % nitric acid at 66C for times ranging from 1/2 to 6 minutes, the etch rate for dingot cores was 3.5 mg/cm²-min. as compared to 4.9 mg/cm²-min. for ingot cores.³⁰

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MCW³¹ investigated the mode of nitric acid attack on alloyed dingot and ingot uranium and found that the acid etched the metal differently. The beta treated alloyed dingot metal was pitted primarily at grain boundaries with little granular attack, whereas, the beta treated ingot metal was deeply pitted due to the dissolution of carbides and other inclusions and small pits were also found within the grains.

The beta heat treatment of alloyed dingot uranium dissolves the major fraction of intermetallic compounds which were precipitated during the alpha fabrication process. A post beta treatment alpha anneal re-precipitates the intermetallic compound U_6Fe and possibly U_3Si . These particles are initially located within the grains; consequently, the acid attack of the alpha annealed alloyed dingot metal is shifted from the grain boundary to the precipitates within the grains. A post beta treatment alpha vacuum anneal at 950 to 1000F for 8 hours increases the etch rate of alloyed dingot metal and preliminary data indicates that alloyed dingot metal receiving this alpha anneal produces better external bonds during canning than for regular alloyed dingot metal.³²

B. Bonding

When uranium cores react with Al-Si during the canning operation a mixture of two or more intermetallic compounds (believed to be primarily USi_3 and UAl_3) are formed on the core surface.³³ Alloyed dingot cores form a higher percentage of the USi_3 -type compound on the core surface than ingot cores at the same canning conditions. The USi_3 -type bond is believed to be inferior to the UAl_3 -type bond because it is: ³¹

1. more brittle as noted from microhardness tests,
2. quite laminar and propagates a crack whereas the UAl_3 -type bond tends to stop a crack,
3. a poorer heat transfer medium than the UAl_3 -type bond by a factor more than three.

With alloyed dingot uranium an additional factor of a slower surface chemical reaction rate may increase the probability of producing a brittle USi_3 layer. Examples of a brittle, cracked compound layer and an essentially nonwet uranium surface on the I.D. surface of alloyed dingot production cores are shown in Figure 19. An example of a feathery tough compound layer formed on the outer surface of a normal ingot core is shown for comparison.

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It was concluded from development data in the summer of 1962 that the compound layer structure of dingot and ingot can be made essentially identical by reducing the silicon content of the duplex furnace AlSi melt from 9.0 to 7.5 per cent and increasing the duplex furnace lead temperature from 616 to 625C. When production canning of alloyed dingot cores was carried out in August and October, 1962, using the new duplex furnace conditions it was found that the desired bonding structure could be produced on dingot cores. However, on certain canning lines it was found for both ingot and dingot cores that fractured internal bonds were being produced even though the normally measured canning furnace and process parameters were within limits.³²

One program to improve the internal surface bonding on dingot cores is to lead plug the bore before inserting the core into the duplex bath. The lead plug prevents the bore from becoming plugged with frozen Al-Si. The lead melts out quickly allowing molten lead to fully preheat the inner surface. This lead plugging technique has been used for canning all CVIN overbore cores and the preheat rate was about twice as fast as by the usual method.

III Irradiation Characteristics

A. Quantity of Dingot Irradiated

The monthly status of "O" and "K" size alloyed dingot uranium being irradiated and the total tonnage discharged is shown in Figure 20. As can be seen, the tonnage of "O" size dingot cores being irradiated leveled out at about 300 tons per month from May, 1961 through April, 1962, while the discharge rate has been averaging about 72 tons/month from January 1961 through June 1962.

B. Dimensional Data

An analysis of Quality Certification and Production Test post-irradiation data shows the following difference between "O" size normal ingot and alloyed dingot fuel element average warp, OD₂ (mid-core diameter) change, and tube filling capacity by shipping data from the feed sites (as shown in Figure 21):

1. Warp characteristics of dingot and ingot material were not significantly different. The average warp values for material shipped in late 1960 and early 1961 are about 1 to 1-1/2 mils low due to a calculation bias on some of the fuel elements.
2. The average OD₂ change for dingot cores shipped in 1960 was about 4 mils larger than for ingot cores but for dingot cores shipped in 1961 the OD₂ change is comparable with ingot elements.

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3. The average tube filling capacity for both ingot and dingot elements show a decreasing trend as the average warp values decrease. The tube filling capacity for dingot cores shipped in 1960 is higher than for ingot cores due to the larger average OD_2 change.

A comprehensive evaluation of dimensional data comparing normal ingot and alloyed dingot uranium is reported by R. R. Bloomstrand.³⁴

C. Failures

1. Failure Types and Rates

The classification of "O" size normal ingot and alloyed dingot failures is shown by failure date, canning date, and shipping date from the feed site in Figures 22, 23 and 24, respectively. In Figure 24, only the virgin lots in which the shipping date can be determined are included, therefore, there are fewer failures plotted in Figure 24 than in Figure 22 or 23. Six of the 26 alloyed dingot failures through May 1962 were classified as core split failures. The theories of split failure mechanism are discussed in Section III, C, 3. The side-hot-spot failures are attributed to core warp or misalignment of the element within the process tube; the internal and end failures to water entry through a defective jacket or weld; and the side other to core warp, misalignment in the process tube, corrosion of the aluminum jacket, or mechanical damage to the jacket during charging.

To compare normal ingot and alloyed dingot fuels as to failure performance, a sequential test designed by Jaech²¹ was used. Only normal ingot material in the same reactor with alloyed dingot elements was used for comparison. Bumpered and non-bumpered elements are handled as two separate sequential tests. The sequential test was designed to demonstrate either that dingot was equivalent to normal ingot elements with 90% confidence or dingot was two fold worse than normal ingot with 95% confidence. A plot of the sequential test by failure date is shown in Figure 25 for the nonbumpered fuel elements. As shown, the sequential test dropped below the lower control limit (where dingot is two fold worse than ingot) in April 1962 after 20 nonbumpered dingot failures and 10 nonbumpered ingot control failures had occurred. There have not been enough bumpered element failures (6 dingot, 3 ingot control) to make a sequential test plot significant.

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In Table I the failures are summarized through May 1962 and in Table II the alloyed dingot and normal ingot element control failures are listed by date with the power, exposure and failure classification. The fuel element performance level, q , of nonbumped dingot fuel was 2.6 times worse than that of ingot control fuel in May 1962.

2. Radiometallurgical Examinations

The first five split failures in alloyed dingot fuel elements were examined by the Radiometallurgy Laboratory in an effort to determine the failure mechanism. As can be seen in Figures 26 through 29, the failures were primarily multiple transverse split failures held together by the aluminum spire.

Since the dingot core lots that sustained the first two split failures also contained external surface cracks, as shown in Figure 17, it was thought that there was an association between the surface cracks and transverse splitting. Therefore, the dingot number stamped on the cores were obtained by chemically stripping the aluminum jacket from the ends of the first failure^{as} and from the entire core on the second failure and the external surface was examined for cracks. No relation, however, could be found between the two failures and external quench cracked cores found during surface inspection.

The findings resulting from examination of the five alloyed dingot split failures by Radiometallurgy and Radiochemistry are summarized as follows:

- a. Extensive water-uranium reaction had occurred around the spires essentially for the entire length of the fuel elements.
- b. The cans were necked down at the site of the transverse cracks but the spires were not necked down beneath the transverse splits, which suggests that the spires were unbonded when the elements parted.
- c. A network of cracks was found on the I. D. surface of most of the cores examined as shown in Figure 27.
- d. Transverse and longitudinal cracks radiated outward from the I. D. surface as shown in Figures 26 and 27.
- e. Continuing water-uranium reaction within the cracks had apparently propagated the cracks until failure occurred as shown in Figure 29.

Table II LIST OF ALLOYED DINGOT AND NORMAL INGOT CONTROL FAILURES

Alloyed Dingt Failures						Normal Ingot Control Failures					
Failure Number	Lot Number	Failure Date	Power KW/Tube	Exposure MWD/T	Failure Type	Failure Number	Lot Number	Failure Date	Power KW/Tube	Exposure MWD/T	Failure Type
1	VL009D	3-25-60	1135	560	Unclassified	1	CL892D	3-21-60	1187	548	Side Other
2	VL012D	5-1-60	1118	278	Side Hot Spot	2	KL485D	7-31-60	1006	478	Side Other
3	VL037D	9-24-60	1055	640	Side Hot Spot	3	KL559D	12-18-60	1207	603	Side Other
4	VL035D	2-21-61	1222	774	Side Hot Spot	4	CL951A	1-13-61	1170	475	Side Hot Spot
5*	OL010D	4-7-61	596	745	Unclassified	5	KL587H	3-20-61	1282	517	Unclassified
6*	OL010D	4-28-61	631	760	Internal	6	ML005H	4-6-61	1252	623	Side Hot Spot
7**	LZ500A	5-1-61	1268	491	Side Hot Spot	7	ML001A	4-22-61	1293	639	Side Hot Spot
8**	LZ500A	5-1-61	1167	450	Unclassified	8	KL608A	4-24-61	1102	542	Side Hot Spot
9*	UZ014A	6-9-61	977	305	Split (Trans.)	9***	KZ064E	8-19-61	1159	795	Internal
10	VZ006B	6-17-61	1171	252	End (Base)	10***	KZ024E	10-10-61	1106	758	Internal
11*	UZ015H	6-24-61	1237	208	End (Cap)	11	KZ083A	12-27-61	1256	750	Side Hot Spot
12*	UZ015H	7-1-61	1172	419	Split (Trans.)	12	KZ152A	3-11-62	1123	469	Side Hot Spot
13	PZ010A	8-31-61	1025	786	Internal	13***	KZ138D	3-12-62	1226	263	End (Base)
14	HZ046B	10-29-61	1139	274	Split (Trans.)	14	KZ142H	5-12-62	1166	627	Unclassified
15	PZ019H	10-31-61	1148	337	Split (Trans.)	15	KZ307A	6-25-62	1168	486	Side Hot Spot
16	HZ036H	12-14-61	1229	780	Side Hot Spot						
17*	UZ057H	1-7-62	1195	208	Split						
18***	HZ043E	1-9-62	1145	253	Unclassified						
19***	VZ032D	1-30-62	1107	976	Split						
20*	UZ060A	3-9-62	1234	289	End (Cap)						
21***	HZ065D	4-16-62	1267	79	Internal						
22	HZ068A	4-20-62	1169	121	Internal						
23***	HZ065D	4-21-62	1179	118	End (Base)						
24*	UZ082A	4-26-62	1183	221	Unknown						
25*	UZ082A	5-1-62	1194	197	End (Cap)						
26	HZ069A	5-8-62	1022	226	End						
27***	HZ060D	6-14-62	1107	533	Unclassified						
28***	HZ060D	6-18-62	1232	600	Side Other						
29*	UZ068B	6-26-62	910	455	Unknown						

Aluminum Component Vendor

Vendor	Number of Failures	
	Dingot	Ingot
'H'	5	4
'B'	3	2
'A'	18	8

*Vacuum-outgassed-before-heat-treatment cores
 **Large grain lot
 ***Bumpered fuel elements

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- f. No evidence of water entry through the welds could be found on the three elements examined for weld defects.
- g. X-ray diffraction measurements on four of the cores measured to date were not abnormal.
- h. The iron content of the first four split cores was not high (less than 200 ppm).

3. Theories of Split Failure Mechanism

Since normal ingot and alloyed dingot uranium are rolled and heat treated by essentially the same process, the sensitivity of alloyed dingot metal to transverse cracking appears to be associated with differences in chemistry. The primary chemical differences between normal ingot and alloyed dingot metal are in the carbon, iron, and silicon content as follows:

	<u>Alloyed Dingot</u>	<u>Normal Ingot</u>
Average Carbon	<30	445
Average Iron	150	110
Average Silicon	105	40

During the quench after salt bath heat treatment, the beta-alpha transformation results in a volume decrease that produces small randomly oriented shrinkage cracks in the center portion of solid dingot core blanks as shown in Figure 15. In normal ingot uranium this volume change occurs primarily at inclusion stringers (mainly carbides) which are oriented in the longitudinal direction near the center of the solid core blank. The majority of the shrinkage cracks are removed during the core drilling operation, but some of the randomly oriented cracks remain on the I. D. surface of dingot cores as shown in Figure 16. Uranium hydrides are also found near the I. D. surface on some of the dingot cores that may act as stress risers. MCW has determined that dingot metal with 180 ppm iron is more brittle than dingot with lower iron content or normal ingot uranium as shown in Figures 13 and 14. In brittle materials less stress is required to propagate cracks.

There are currently two theories for explaining the mechanism of transverse cracking in alloyed dingot fuel elements:

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- a. Water reaction in the cracks (or hydrides) wedges the metal apart from the inside surface outwards to the exterior surface by a build-up of voluminous uranium oxide. This mechanism appears to be the most likely of the two theories. However, it does require water entry to a poorly bonded spire and no source of water entry has been found on any of the three split dingot failures examined for water entry. The amount of water reaction around the spire (up to 1/8 inch in depth) for essentially the entire length of the core suggest that extensive water reaction could not have occurred after discharge and storage according to data reported previously by Evans.³⁶
- b. High tensile residual stresses resulting from the Al-Si canning process. Joseph³⁷ has measured longitudinal and circumferential tensile residual stresses on the I. D. surfaces of Al-Si canned cores (triple-dip process) in the order of 100,000 psi. These high stresses are the result of the cold water quench after canning which quickly cools the outer surface and allows the inner surface to cool slowly. However, if a dingot core were to fail by these stresses propagating the shrinkage cracks or propagating cracks from hydride stringers, the element should fail suddenly at one location with very little water reaction between the spire and inner surface of the core. Also at operating temperatures within the reactor it is expected that any residual stresses would be removed by annealing.

If an ingot core were to split, one would expect that the split would be longitudinal since the shrinkage voids are associated with longitudinal impurity stringers.

IV. Overbore Fuel Program

In January 1961, 20 overbored (550-mil) graphite channels and process tubes were installed in C Reactor. This test facility was expanded to 42 tubes in September 1961, and to 62 tubes in December 1961. The large diameter CVIN cores (1.883" O.D., 0.452" I.D.) required for the overbored tubes were fabricated by the following methods:

1. An initial eight tons of CVIN cores were fabricated in November 1960, from low hydrogen alloyed dingot uranium that was alpha extruded into tubes by MCW and tube straightened, hollow core blank beta heat treated, and machined by NLO.

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2. A second order of 14 tons of CVIN cores was fabricated in December 1960 from rolled ingot rod, solid core blank heat treated, and machined by NLO. The rods were removed from the No. 2 rolling stand as an oval rod (normal 1.5-inch rods pass through 6 rolling stands and are round).
3. Subsequent dingot CVIN cores (62 tons) supplied to HAP0 were fabricated from alloyed dingot metal that was alpha extruded into tubes, hollow core blank heat treated, and machined by MCW. Also, 42 tons of the 62 tons received were vacuum outgassed after beta heat treatment to remove hydrides from the inner and outer surfaces.
4. All subsequent overbore core production after October 1962 was scheduled to be fabricated from rolled ingot metal.

Above standard canned fuel element quality was achieved through adoption of process innovations such as the use of an ultrasonic test to detect internal and external longitudinal cracks; insertion of lead plugs in the cores to accelerate preheat in the duplex canning bath and to improve internal bonding; use of pressurized quench after canning to suppress braze porosity; and use of UT-4 testers to detect and reject elements with defective Al-Si braze closures.

Examination of some 33 columns of CVIN fuel elements after irradiation has shown that the average warp and tube filling capacity is comparable to standard I & E fuel elements and shows essentially the same tendency to become "barrel" shaped during irradiation. The dingot cores tend to grow larger in diameter than do the ingot cores by about 2 or 3 mils.

Approximately 4,300 overbore fuel elements had been discharged as of the first of December 1962 at exposures ranging from 200 to 1,000 MWD/T, with the majority of the tubes in the range of 600 - 900 MWD/T. Three failures had occurred in alloyed dingot cores and four in ingot cores as follows:

<u>Failure Number</u>	<u>Lot Number</u>	<u>Failure Date</u>	<u>Power KW/Tube</u>	<u>Exposure MWD/T</u>	<u>Failure Type</u>
1	KX-010	1-16-62	1492	691	Side Other - Missing rail
2	KX-010	4-16-62	1439	671	Split - Transverse**
3	KX-010	4-16-62	1439	671	Split - Transverse**
4	KX-010	4-16-62	1439	671	Split - Transverse**
5*	WP-004	8-29-62	1243	640	Split - Transverse
6*	WP-003	9-3-62	1201	710	Side Other - Broken rail
7*	WP-004	9-6-62	1265	661	Split

*Alloyed dingot core

**Three ingot failures in one tube after experiencing severe reactor flux cycling.³⁸

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Because dingot uranium fuel elements proved to be generally more rupture prone in the Hanford Reactors, the exposure of dingot overbore fuel was reduced to 75 percent of variable (800 MWD/T) until the inventory of alpha extruded dingot overbore cores could be exhausted. Alpha rolled ingot cores will be used to meet future overbore fuel requirements.

APPROVED:

Manager

Fuels Engineering Operation

ISSUED:

Engineer

Product Engineering

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Figure 1. INGOT AND DINGOT URANIUM FLOW SHEET

Figure 2. NORMAL INGOT AND DINGOT URANIUM
AVERAGE CARBON CONTENT

Average Carbon Content, P.P.M.

600

500

400

normal ingot

300

200

100

dingot

0

DD-500-001 (11-50)
U.S. GOVERNMENT PRINTING OFFICE

1960.

1961

1962

Month Analyzed by Feed Site

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37

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Alloyed Dingot Uranium- 5 ppm C
10 ppm N

Normal Ingot Uranium-442 ppm C
41 ppm N

Figure 3. Transverse Normal Ingot and Alloyed Dingot
Core Sections Showing Inclusions (Primarily
Carbides). 250X

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Figure 8. AVERAGE GROWTH INDEX (G_2) FOR NORMAL INLET
AND ALIGNED INLET CORES

Normal Inlet Growth Index, G_2

Aligned Inlet Growth Index, G_3

Figure 3. AVERAGE 200 TIMES EXPOSURE CONTRASTING FOR NORMAL EXPOSURE AND ALLEGED BINGSON

SEP 1962 (11-60)
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1960

1961

1962

1961

1961

1961

1961

Figure 11. AVERAGE GRAIN SIZE OF NORMAL INGOT AND ALLOCATED DINGOT CORES MEASURED AT HMPO VS SHIPPING DATE FROM FEED SITES

SHIPPING DATE FROM FEED SITES

- Note 1 - Large grain dingot cores believed due to quench conditions. Controls initiated by Jan. '61.
- Note 2 - Large grain normal ingot cores due to large grain size on one end. Believed due to a quench problem.
- Note 3 - Reason for apparent increase in dingot grain size believed due to increased rolling temperature of dingot rods in Aug. '61.

100-100000

100-100000

1000

1000

1000

1000

1000

Alloyed Dingot Uranium No. 7159A

Normal Ingot Uranium No. 82900

Figure 15. Longitudinal section through the center of an alloyed dingot and a normal ingot beta heat treated solid core blank showing shrinkage voids or cracks resulting from the beta-alpha transformation. 3X

Small crack at I.D. Surface

Hydrides near core I.D.

Figure 16. Transverse sections through a vacuum-outgassed-before-heat-treatment alloyed dingot core showing a small crack at the I.D. surface and hydrides near the I.D. surface. Dingot number UZ-9230A. 250X

Quench-cracked Normal Ingot Core Containing 606 ppm Fe.
Ingot No. R-50523

Quench-cracked Alloyed Dingot Core. Dingot No. UZ-5657B

Figure 17. Surface appearance of quench-cracked normal ingot and alloyed dingot recovered cores. Cracking during beta heat treating of the ingot metal was attributed to high iron content (>500 ppm) and in alloyed dingot metal was believed due to corrosive attack of core blanks during damp storage prior to heat treatment which produced cracks during heat treatment. $3/4X$

Figure 18. VISUAL SURVEIL INDEX (VSI) RATES AT HAPC FOR NORMAL AND INJURED COCKS

Visual Survey Index (VSI) Rates

1960-1961-1962 (1960-1962)
ATC 1000, HAPC HAZ

Normal

1961

1962

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Figure 19. EXAMPLES OF TOUGH AND BRITTLE COMPOUND LAYERS
AND NONWET URANIUM

I.D. surface of an alloyed dingot core that is essentially nonwet except for the brittle layer near the center. 250X

I.D. surface of an alloyed dingot core showing cracks in a brittle compound layer believed to be primarily USi_3 . 250X

O.D. surface of a normal ingot core showing a typical tough feathery compound layer believed to be primarily UAl_3 . 250X

[REDACTED]

1968

1969

Month

1970

1971

DECLASSIFIED

11-7350

-1-

[REDACTED]

UNCLASSIFIED

Figure 22. '01 Side Normal Ingot and Allowed Ingot Produced by Various Days

As of 5/31/62

- ☒ - Side Hot Spot
- ☐ - Side Other
- ☒ - Side Split
- ☐ - Side
- ☐ - Internal
- ☐ - Unclassified
- ☐ - Unknown
- ☐ - Ingot Control

11-11-66

15-00000

Figure 24. '0' SHOT FOUND. DEFECT AND ALLOYED DEFECTS. THICKNESS
SHOOTING RANGE. 1959-1960

100-73160

100-73160

100-73160

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VI = Response Index = $E_p \cdot p_p \cdot p_p \cdot 2$

VI = $\frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2}$

VI = $\frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2}$

VI = $\frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2}$

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VI = $\frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2} \cdot \frac{1}{2}$

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11-73-14

Figure 26. First transverse split failure in an additive dingot fuel element. About a 1/2" section is missing from the center of the element. Note transverse and longitudinal cracks radiating from the I. D. Dingot No: UZ-5751B
Exposure: 305 MWD/T - Power: 44 KW/FT.

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HM-73149

DECLASSIFIED

1.5X

Figure 27. Longitudinal sections from the second transverse split failure in an additive dingot OIIIIN fuel element showing a network of cracks on the I.D. surface and transverse cracks radiating from the I.D. Lot UZ-015H.
Exposure - 419 MWD/T - Power - 53 KW/FT.

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THIRD TRANSVERSE SPLIT FAILURE. LOT HZ-046B
EXPOSURE - 274 MWD/T. POWER - 51 KW/FT

FOURTH TRANSVERSE SPLIT FAILURE. LOT PZ-019H
EXPOSURE - 337 MWD/T POWER - 50 KW/FT

Figure 28. Appearance of the third and fourth transverse split failures in additive dingot OIIIN fuel elements.

2/3 X

DECLASSIFIED

1.7 X

Figure 29. Fifth split failure in an additive dingot OIIIN Fuel Element. In the longitudinal section note how the reaction has proceeded outward from the I.D. until the core cracked through to the O.D. necking the aluminum can wall. Lot UZ-057H Exposure - 208 MWD/T. Power - 54 KW/ft.

DATE

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9 / 26 / 94

END