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**N-REACTOR LITHIUM-ALUMINATE
TARGET ELEMENT PROCESS
DEVELOPMENT AND FABRICATION**

BY
C. H. SHAW

**N-FUELS ENGINEERING OPERATION
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DECEMBER 30, 1967

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TARGET ELEMENT PROCESS
DEVELOPMENT AND FABRICATION

By

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Preparation Date: June 1967

Date of Issuance: 12/30/67

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TARGET ELEMENT PROCESS
DEVELOPMENT AND FABRICATION

C. H. Shaw

ABSTRACT

Fabrication of LiAlO_2 target elements for the N-Reactor Coproduct Demonstration Program required development of a suitable fabrication process consistent with installation of a minimum production facility on a limited time schedule. The most advanced manufacturing process developed and used within the facility and schedule constraints is described in detail. Major problems encountered in obtaining required component materials and development of the fabrication process are discussed and analyzed for impact on future programs utilizing LiAlO_2 target elements.

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INTRODUCTION

The coproduct target element fabrication process used consisted of a typical sintered product process coupled with cladding and support hardware attachment operations to adapt the sintered product for use in the reactor. The facility and equipment utilized for production was minimized due to the short program duration. This minimization of capital expenditure and the limited time schedule were in many cases more restrictive on the process selected than established product criteria. The fabrication process utilized for a major portion of the demonstration program has been proven capable of producing a product within established criteria⁽¹⁾ with practical manufacturing operations. The process could, however, be refined in many areas from both an operating efficiency and control standpoint by investing additional process engineering development time and capital expenditures for the overall facility.

Quality and reproducibility of input LiAlO_2 powder characteristics was the most significant problem encountered in the program. Development work to define and control the processing parameters necessary to produce LiAlO_2 powder with consistent characteristics should be of first priority in any continued irradiation program utilizing LiAlO_2 targets. Supply of other essential components used in the program was more a matter of establishing reliable fabrication routes rather than basic development of material processes.

MANUFACTURING PROCESS

The coproduct target element manufacturing process was basically made up of batching operations. Individual components were fabricated in lots and assembled into the finished element. For purposes of process control, each target assembly was considered a unique lot.

GENERAL PROCESS DESCRIPTION

A process flow diagram for coproduct element fabrication is shown in Figure 1. Target element details are shown in Figure 2. The LiAlO_2 powder was pebble milled for particle sizing, appropriate binders were added to increase green strength, and the powder was then pressed into pellets. The green pellets were preheated for binder bakeout followed by sintering at 1325°C for 14 hr. The sintered pellets were centerless ground, squared, and cut to stack length for assembly into aluminum cans. The aluminum clad assemblies were hydrostatically sized to the ceramic outside diameter, straightened, centerless ground, and assembled into the zircaloy cladding. The zircaloy clad assemblies were support welded and autoclaved followed by helium leak testing immediately prior to assembly with driver fuel. Each of the manufacturing process steps illustrated in Figure 1 is discussed under "Fabrication Processes."

The basic process for fabrication of sintered pellets was essentially that used for manufacture of most ceramic products. The process used did not provide for close dimensional control of the sintered product; however, sufficient development time was not available prior to production startup to optimize in this area. The decision to use secondary finishing operations was dictated by overall schedule commitments. Further refinements of the sintering process are advisable to minimize dimensional variation of the product in a sustained operation.

CO-PRODUCT TARGET FABRICATION PROCESS

FIGURE 1. Coproduct Target Fabrication Process

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FIGURE 2. Cross Section of Coproduct Target Element

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The assembly and cladding operations used were not unusual for nuclear fuel. Glovebox assembly of the pellets into aluminum was required to minimize moisture pickup in the ceramic. The major technical consideration in the assembly area, other than physical integrity, was that the moisture level in the finished product had to be maintained less than 150 ppm. This moisture level was attained by drying the ceramic at 800 °C. The pellets were then immediately transferred at temperature to a controlled atmosphere (argon) glovebox for assembly in aluminum. The ceramic was not exposed to room air following the drying step. This particular portion of the assembly process dictated a batch-type of operation. The glovebox operation was not limiting to production throughput in any manner when sufficient inventories of sintered product existed to process reasonable size batches.

PROCESS CONTROL PROCEDURES

Three basic documents were used for information to control the fabrication process. These documents discussed

- Operating Procedures⁽²⁾ which were a combination of process and equipment operating instructions. The procedures were utilized directly by operating personnel and were, therefore, the most restrictive.
 - Quality Control Standards⁽³⁾, which detailed and limited those process and equipment parameters of major importance to product quality. The standards were used for auditing purposes by Quality Control personnel, and were always less restrictive than the Operating Procedures.
 - Engineering Specifications⁽⁴⁾ which define those process requirements necessary to assure conformance to product criteria. The specifications were a combination of product and process requirements. The specifications were also used as a base for both the Quality Control Standards and the Operating Procedures.
- [REDACTED]

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Control requirements for coproduct target elements called for development of a material identification system to insure that the processing and materials history for each element could be traced. This requirement was met by the use of an alpha-numeric identification number assigned each element. This identification number plus recorded manufacturing processing details associated with a given element were similar to those used for most nuclear fuel elements. This system is described in detail in Appendix A.

FACILITIES AND EQUIPMENT

The equipment and facilities to produce the coproduct target elements were installed with minimum capital expenditure.^(5,6) As stated under "GENERAL PROCESS DESCRIPTION," the limited time schedule dictated by the demonstration program required development of a practical compromise between installed equipment and the fabrication process used. The equipment used, however, performed quite satisfactorily and could be used for sustained operations. It is possible, with continued process development work, that the overall length of the production line could be shortened with resultant decreases in the number of equipment items required. This is not to say that the overall facility floor space would be reduced or that installed equipment cost would be reduced. To the contrary, a reduction of process steps, particularly in the pellet sizing area, would require the use of more sophisticated equipment to obtain reproducible control of the sintered product dimensions. A detailed listing of the major equipment items is included in Appendix B.

The most significant problems with fabrication equipment were related primarily to the characteristics of lithium aluminate and the product variability encountered. The aluminate powder is an abrasive and, therefore, all moving parts on the process equipment must be protected. The abrasive effects of the powder were most evident on the pellet press, press tooling,

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blender bearings, grinder bearings, etc. Excess equipment capacity in these areas was necessary to allow for the extensive preventative maintenance required to protect critical equipment components.

Sintering furnace performance is a continuing problem due to lithium boil-off during the sintering operation. The lithium reacts with the furnace brick and globar heating elements dictating an average replacement of globars every seven weeks and furnace brick every three to four months. Attempts were made to improve globar life by pressurizing each unit with air. The air flow through the furnace provided some protection for the heating elements, but created significant temperature differences across the furnaces with resultant variation in sintered pellet density. Scaling the furnaces did not, therefore, prove practical with the installed furnace designs. Engineering effort was then directed at testing and procurement of components least affected by the lithium. A simple change from the A. T. Standard type of globar to the S. G. spiral type provided a 300% increase in life.

Sintered densities were controlled $\pm 5\%$ from specified nominal (78% of theoretical). This amount of variation, however, created numerous problems with the pellet sizing equipment. Pellet reaction to abrasive saw blades and grinder belts is dependent on density and the amount of variation allowed in the product required tailoring of grinding equipment to each sintering lot; i.e., a different grade of blade was required for sawing 32% dense pellets than the one used for 74% dense pellets.

COMPONENT MATERIALSLITHIUM-ALUMINATE

Lithium-aluminate (LiAlO_2) is produced by reacting lithium-carbonate (LiCO_3) and alumina (Al_2O_3) at temperatures between 750 and 1000 °C. LiAlO_2 has two crystallographic forms. The low form, α , transforms irreversibly to the high form, γ , above 600 °C with an accompanying 30% volumetric increase. The kinetics of the transformation increase with increasing temperature and the transformation occurs fairly rapidly at 900 °C. The reaction can be accomplished by heating stoichiometric quantities of lithium-carbonate and alumina either as pressed compacts or powder mixtures.

Chemical purity of the aluminate is, of course, largely dependent on carbonate and alumina purity and use of clean processing equipment. The chemical purity of the final product is also dependent on the completeness of reaction and stoichiometry of the carbonate plus alumina. Most of the aluminate used for the demonstration program had substoichiometric lithium contents with residual carbonate and alumina. Residual carbonate is undesirable if the material is subsequently heated above the reaction temperature due to reaction of the carbonate producing a partial pressure of CO_2 . Residual alumina is also undesirable due to effects on powder sinterability.

Control of the isotope ratio ($\text{Li}^6/\text{Total Li}$) in the lithium carbonate feed material was accomplished by blending large batches of lithium hydroxide monohydrate ($\text{LiOH} \cdot \text{H}_2\text{O}$) to the required ratio. The hydroxide was then converted to carbonate and reacted to aluminate. This approach to isotope ratio control was used to simplify vendor certification procedures from hydroxide to aluminate since control of processing temperatures and product stoichiometry assured a given isotope level in the aluminate with

given hydroxide input. The problems encountered in establishing the supply of LiAlO_2 with required isotope level are discussed below in more detail.

LiAlO_2 Supply

Lithium-aluminate was produced, prior to HAPD usage, in small quantities by only one U.S. concern. This situation still exists. Other concerns expressed interest, but were not willing to perform the process development work and facility modifications required to produce material for a limited application. The primary supplier's experience in producing LiAlO_2 was limited to an occasional 200 pound lot which was offered to the ceramics industry for a specialty glaze. Lithium content varied widely and impurities were controlled only as they affected the color of the product.

The material for the demonstration program represented more than a 100-fold increase in annual production and an entirely new magnitude of chemical control⁽⁷⁾ in the product. The aluminate producer was not required to establish or maintain control of isotopic level except by segregation of the hydroxide feed material with various isotopic contents. Vendor performance in maintaining total lithium content in the product was quite good on all material produced. Control of specified impurity levels was more than adequate except for two elements, boron and fluorine.^(8,9)

The universally accepted method of analyzing for lithium is by flame photometry. All lots of aluminate were analyzed and reported by both the vendor and Battelle-Northwest Chemical Analysis Section. While absolute agreement on any one lot was rare, the averages of values reported for all lots was surprisingly close considering the inherent lack of precision of the analytical method.

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No quantitative measure of boron content was available on initial production of aluminate. A spectrographic analysis for boron on a semiquantitative level was developed by Douglas United Nuclear Spectrographic Laboratory, and boron was found to occur sporadically in quantities up to 200 ppm. This level of contamination is subsequently reduced at least 50% by boil-off during the sintering operation. Boron contamination existed due to a cross-contamination problem in the reaction process. The problem was eliminated by emphasizing the need for complete separation of the aluminate from boron containing materials and contaminated equipment.

The practical limits on fluorine content are not known. High fluorine content is potentially more serious than high boron, which only affects fuel reactivity and product yield. Fluorine can affect fuel integrity under irradiation and free fluorine or volatile fluorine compounds in the product separations operation could prove detrimental to some items of equipment. (10) Based on the above general considerations, a limit of 25 ppm fluorine was established for the aluminate. Again, as in the boron case, analytical methods had to be developed to perform fluorine analyses. Due to delays in obtaining analyses and the shortage of available powder, a number of powder lots were used which contained fluorine in excess of 25 ppm. This material was used because: (1) preliminary evaluation of product separation operations indicated that no free fluorine and very few, if any, fluorine compounds were released; and (2) a review of fundamental fluoride chemistry showed that the fluorine content of the pellets should be significantly reduced during sintering. Subsequent tests have shown that the fluorine content is reduced by at least a factor of 10 by sintering. The source(s) of fluorine has not been determined, but it is quite possible that a significant amount existed in the hydroxide used, and the reaction process was not performed at high enough temperatures to reduce the content by boil-off.

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Isotope Control

Procedures were required to establish and then control specified quantities of the Li^6 isotope per unit of finished target element volume. Early in the program, isotopic ratio of the aluminate was adjusted by blending natural lithium values with values of a lower Li^6/Li^7 ratio than was desired in the finished product. Total lithium and Li^6 ratio analyses were made on each batch of aluminate received and materials proportioned to yield the desired Li^6 content per unit target volume at 78% of theoretical density. This powder blending approach did not prove practical in that batch sizes were small and the inherent difficulties in mass spectographic determination of the Li^6/Li weight ratio dictated use of maximum batch sizes to minimize the number of analyses required to certify the product. Isotopic ratio adjustment was therefore accomplished by solution blending large batches of lithium hydroxide to the desired level.

Adjustment in the hydroxide was believed possible since subsequent processing operations should not vary the ratio. The control problem was then reduced to control of total lithium as affected by the quantities of Li_2CO_3 and Al_2O_3 used in the reaction to LiAlO_2 , the boil-off losses during sintering, and the density of finished pellets.

The isotope control problem was primarily concerned with analytical ability to provide accurate analyses of the product at various points in the fabrication process. The level of Li^6 can be obtained directly by the combination of chemical and mass spectographic analyses or indirectly by reactor determination of reactivity versus standards. The control requirements established by performance criteria are detailed in Table I.

TABLE I. Allowable Variation of Average Li^6 Content from Specification Nominal

Li^6 Control Base	Base Case (Nom. = 6.07 mg Li^6/cm^3 of LiAlO_2)	Spike Case (Nom. = 3.68 g Li^6/cm^3 of LiAlO_2)	Poison Case (Nom. = 12.26 mg Li^6/cm^3 of LiAlO_2)
Individual Targets	$\pm 10\%$	$\pm 15\%$	$\pm 10\%$
Average of all Targets in a Reac- tor to Column	$\pm 5\%$	$\pm 7 \frac{1}{2}\%$	$\pm 5\%$
Average of all Targets in the Reactor	$\pm 2\%$	$\pm 3\%$	$\pm 2\%$

Chemical and mass spectrographic determination of the $\text{Li}^6/\text{LiAlO}_2$ weight ratio involves separate determination of the Li^6/Li weight ratio (mass spectrographic) and the Li/LiAlO_2 weight ratio (flame spectrometry). Each of these analyses is considered accurate to $\pm 7\%$. The $\text{Li}^6/\text{LiAlO}_2$ weight ratio determined by the product of above analyses is considered accurate to $\pm 10\%$. The accuracy of the $\text{Li}^6/\text{LiAlO}_2$ weight ratio determination could be improved by either improved accuracy on individual Li^6/Li and Li/LiAlO_2 analyses, or by multiple analyses on a given sample. The Li^6/Li mass spectrographic analysis is considered good at an accuracy of $\pm 5\text{--}7\%$ due to the low Li^6 content (3 wt% Li^6 in base case material). The Li/LiAlO_2 analysis is complicated by the time dependency of the analysis due to lithium reaction with container materials. The Li/LiAlO_2 analysis is usually accurate to $\pm 5\%$; however, the wide range of experienced values indicates the use of $\pm 7\%$ for higher certainty.

Reactivity tests (305 Test Reactor used for all testing) were used to relate a LiAlO_2 reactivity measurement to the powder $\text{Li}^6/\text{LiAlO}_2$ weight ratio. Reactivity measurements for test LiAlO_2 samples were obtained by comparing each sample to

a LiAlO_2 standard of known reactivity. The reactivity value obtained was then compared to a curve of reactivity versus Li^6 content to obtain the $\text{Li}^6/\text{LiAlO}_2$ weight ratio. Three such curves are shown in Figures 3, 4, and 5. The curves shown were used to test aluminum subassemblies, zircaloy clad assemblies, and input powder, respectively. Within curve accuracy, the only variable was the reactivity test measurement. A Li^6 value (assuming correct curve) can therefore be obtained within $\pm 1/2\%$. The curves were, however, generated by multiple analyses and contain inherent inaccuracies based on these analyses. Accuracy levels for the Li^6 determination for the various powder cases (base, spike, and poison) are detailed in Table II.

TABLE II. Accuracy--Isotope Determination

Item Associated with Use of Li^6 Versus Reactivity Curves	Limitations of Accuracy (a)
<u>Subassembly and Target Curves (Figures 3 and 4)</u>	
1. Li^6 values (ordinate of curves) determined by multiple analyses.	$\pm 3 \frac{1}{2}\% \text{Li}^6$
2. Reactivity values (abscissa of curves) determined by multiple analyses.	$\pm 1/4$ inhour or $\pm 1\% \text{Li}^6$ (Base & Poison Cases) $\pm 1/2$ inhour or $\pm 2 \frac{1}{2}\% \text{Li}^6$ (Spike Case)
3. Combination of Li^6 values and reactivity values of 1 and 2 above to obtain curve data points.	$\pm 3 \frac{1}{2}\% \text{Li}^6$ (Base & Poison Case) $\pm 4 \frac{1}{2}\% \text{Li}^6$ (Spike Case)
4. Individual sample reactivity measurements (305 Reactor drift test from standard). Note: This is accuracy of comparison between reactivity determined Li^6 values; that is assuming curves are correct.	± 0.1 inhour or $\pm 1/2\% \text{Li}^6$

TABLE II. (Contd)

Item Associated with Use of Li^6 Versus Reactivity Curves	Limitations of Accuracy ^(a)
<u>LiAlO_2 Powder Curve (Figure 5)</u>	
5. Use of reactivity test value to assign <u>real</u> Li^6 content to material represented by sample.	$\pm 4\% \text{Li}^6$ (Base & Poison Case) $\pm 5\% \text{Li}^6$ (Spike Case)
6. Li^6 values (ordinate of curve) determined by multiple analyses.	$\pm 2 \frac{3}{4}\% \text{Li}^6$
7. Reactivity values (abscissa of curve) determined by multiple analyses.	$\pm 1/2$ inhour or $\pm 5 \frac{1}{2}\% \text{Li}^6$
8. Individual sample reactivity measurements (305 Reactor drift test from standard). Note: This is accuracy of comparison between reactivity determined Li^6 values; that is assuming curve is correct.	± 0.1 inhours or $\pm 1/2\% \text{Li}^6$
9. Use of reactivity test value to assign real $\pm 6\% \text{Li}^6$. Li^6 content to material represented by sample.	$\pm 6\% \text{Li}^6$

(a) These $\pm$ values correspond to $\pm 3 \sigma$ limits.

The isotope control procedure developed, utilizing the analytical methods required with inherent inaccuracies, is charted in Figure 6. As-received powder was sampled for total Li and Li^6 analyses and reactivity tested to establish the $\text{Li}^6/\text{LiAlO}_2$ ratio. The purpose of these tests was to double check supplier lithium analyses and to detect any gross contamination by impurities of concern. The powder was also reactivity tested after sintering to establish the exact $\text{Li}^6/\text{LiAlO}_2$ value to be assigned the sintered product from that powder lot. This approach to powder certification provides maximum protection against a major material commitment prior to quantitative determination of quality.

FIGURE 3. Subassembly Li^6 Content Versus 305 Reactivity Results-Coproduct LiAlO_2

FIGURE 5. LiAlO₂ Powder Li⁶ Content Versus 305 Reactivity Results

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FIGURE 6. Isotope Control Procedure

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ALUMINUM CLADDING

Aluminum was chosen for an intermediate cladding because it is impervious to hydrogen isotopes at operating temperatures, is compatible with other target components, and is fusible at the extraction temperature which permits automatic release of the gaseous irradiation products.

Aluminium cladding components were of no particular problem. The use of 1100 alloy tubing caused some delays in procurement because of a long term industry trend to the use of stronger alloys. Once a supply had been established, however, no further problems occurred in meeting schedule requirements.

The capability of impact forming of cans was established early in the program. The impact route proved adaptable to the length-wall thickness requirements (12.9 in. long x 0.090 in. wall) and provided significant cost reduction over the tubing double-end closure alternative.

The need for nondestructive testing of end caps was further emphasized by defects detected during the autoclave sizing process described in more detail under "FABRICATION PROCESS DETAILS." A typical end cap from one of the autoclave failures experienced is shown in Figure 7. Metallographic examination showed the defect to be an oxide inclusion not discovered by ultrasonic testing at the billet stage of processing.

ZIRCALOY COMPONENTS

Cladding

The zircaloy cladding dimensions were established by coolant flow requirements (outside diameter) and structural strength at operating conditions (wall thickness). In the early stages of component design, it was necessary to resolve the question on the relative advantages of free standing and internally supported (aluminum and ceramic components) cladding. The free standing

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FIGURE 7. *Defective Aluminum End Cap*

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cladding concept provided operational advantages in that generous tolerances and allowances could be allowed on the internal components with no compromise of the zircaloy cladding integrity. In addition, the heavier clad provided greater freedom in methods selected for welding support hardware and end closures. Use of thin-walled tubing required control of the ceramic pellet and aluminum assembly dimensions to minimize clearances between the aluminum subassemblies and zircaloy cladding for adequate support of the zircaloy. Assembly and finishing operations are therefore considerably more critical for the nonfree standing cladding.

The limiting wall thickness for free standing clad of design outside diameter (1.335 in.) was calculated to be approximately 0.080 in. Empirical determinations of the limiting thickness showed it to be approximately 0.100 in. due to ovality effects on stress distribution. The limiting wall thickness for nonfree standing cladding was empirically established by limiting tube collapse over a 0.120 in. unsupported length (differential length between aluminum subassemblies and zircaloy cladding to accommodate differential thermal expansion) when exposed to autoclave conditions of 400 °C and 1500 psia. A wall thickness of 0.040 in. nominal was selected on the basis of these tests.

The unit cost advantages of thinner wall tubes for target cladding persist until wall thickness becomes less than 3% of the tube outside diameter (approximately the ratio of minimum wall thickness target cladding). The selection of the cladding concepts to be used was therefore made by a comparison of the additional amount of zircaloy required for free standing clad versus the estimated additional operational cost associated with the nonfree standing clad. This comparison showed that a saving of approximately \$500,000 could be made by using thin wall

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tubing if suitable assembly and finishing processes could be developed to obtain the 0.006 to 0.010 in. diametral clearance requirement between the aluminum subassemblies and the zircaloy cladding. The assembly process was proven feasible and the thin wall cladding route was selected for the program.

Procurement of zircaloy cladding components proved to be one of the major problems in meeting the demonstration program schedule. Procurement lead times for new tubing, produced by tube reducing extruded blanks to final size, proved to be approximately 12 months. The immediate cladding supply problem was resolved by reworking available excess C and K Reactor process tubes. Three methods of reducing tube diameter were employed (wall thickness held constant--outside diameter reduced from approximately 1.800 in. to 1.340 in.). A majority of the tubing was sized by multiple pass tube reducing. In addition, some 20% of the tubing was sized by free-sink drawing and single pass plug drawing (10% each route). All material was annealed and straightened after the sizing operations.

Two major quality problems were encountered with the cladding: (1) folds on the tubing outer surface which are believed to have been caused by excessive reduction per pass in tube reducing extruded blanks to reactor process tube dimensions,⁽¹¹⁾ and (2) a pronounced tendency to warp when stress relieved in the final autoclaving step.⁽¹²⁾

The fold problem was first discovered and identified as such when a leaker was detected after autoclaving. The zircaloy clad did not appear deformed and the leak may not have been detected except for a continued weeping which stained the exterior of the cladding. Location of the leak was remote from supports or end welds, and could not be attributed to any deviation in target fabrication processes. The clad of the failed target was sectioned and examined to identify the origin of the

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defect. A second, smaller, defect about 4 in. axially distant from the point of water entry was included in the examination. The attached series of photomicrographs (Figures 8 through 11) illustrate the nature of this defect as well as a large number of similar defects in the cladding which have been found at receiving inspection. The high degree of axial alignment of similar visual defects plus the "worked" appearance, Figure 10b, of the metal adjacent to the discontinuity, suggests a defect originating early in the metallurgical history of the material. Possible causes are defects in the extruded tube blank or too-severe reductions on the tube reducer resulting in pinching.

All of the tubing to be reworked to target dimensions was tested using a mass-spectrometer type helium leak detector. Tube ends were plugged and the tube was evacuated by a leak detector. A helium filled shroud was passed along the tube. As the shroud covered a leak, the detector would produce a signal and the leak location was marked. The defect section of the tube was later cut out. Remaining sound sections over 6 ft long were drawn to size.

Ultrasonic testing was originally specified for inspection of the redrawn tube. Sensitivity was to be calibrated to a standard defect 0.050 in. long by 10% of tube wall thickness. An ultrasonic tester was assembled and calibrated to the standard defect size. At this level of sensitivity both extruded and redrawn material invariably yielded a defect signal. Zygo and dye penetrant inspection of suspect areas failed to confirm tester results, and it was not possible with intensive metallographic examination to define any flaws in the tube wall. Measurements of surface finish of both free-sink redrawn and extruded tubes showed that surface variations of the same order as the standard defect were causing spurious defect signals. At this point, the requirement for ultrasonic testing was removed; and a combination of 100% visual inspection of incoming cladding and helium leak testing of completed targets was initiated for control of material quality.

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Neg 467-5147-A

5X

FIGURE 8a. *Outer Surface of Clad at Point of Water Entry*

Neg 467-5147-B

5X

FIGURE 8b. *Inner Surface of Clad at Point of Water Entry*

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Neg 467-5148-A

250X

FIGURE 9a. Start of Larger Defect Area as Found by Polishing from Bottom of Figure 8a

Neg 467-5148-F

50X

FIGURE 9b. Progressing Further into Defect. About 5 mils from Figure 9a

Neg 467-5148-C

50X

FIGURE 10a. *Approximately at Center of Visible Defect
on Right of Figure 8a*

Neg 467-5148-G

250X

FIGURE 10b. *Root of Defect in View of Figure 10a. Grain
Size and Orientation Show Defect Existed Prior
to Final Drawing Operation*

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Neg 467-5148-D

50X

FIGURE 11. Water Path

The definitive testing method for clad integrity is the helium leak detector. The optimum application of this technique is to closure-weld a helium-backfilled target under helium atmosphere and immediately test for leaks. In practice, some time must elapse between closure welding and testing. There was a certain amount of material in-process between stations because supports were welded on the target, prior to testing, to insure that support welding had not compromised cladding integrity. Targets which had not been tested within 48 hr of closure welding were, in the interest of increased leak test reliability, held under vacuum for 30 min and then exposed to helium atmosphere at ambient pressure for 15 min immediately prior to testing.

Examination of the path of water entry through the zircaloy clad points to the possibility that the component may have been helium-tight when leak tested. The combination of hydriding at the base of the fold plus the strain, induced by external

pressure and the internal assembly clearance, could extend the notch the final small increment required to produce complete penetration. A final repressurization and leak test after autoclaving was initiated to make the detection of failed targets less dependent on immediate visual inspection after the targets are removed from the autoclave. A total of 17 defective target elements were detected in this manner.

A severe problem with zircaloy clad target assembly warp was experienced during initial stages of production and, to some degree, all during the program. Initially, this warp problem was attributed to stress relaxation during autoclaving when cold straightening or similar operations were performed as a finishing step on the zircaloy tubing. Use of hot straightening and/or annealing steps following straightening effectively reduced the component warp problem although some warp of the assembly continued to occur with autoclaving.

Support Hardware

The first support design for coproduct ceramic targets was an extrapolation of the "buggy-spring" support used on the inner fuel of Mark I fuel assemblies. Proportional thickness of the zircaloy-2 strip spring material was 0.026 in., resulting in a 0.023 in. maximum thickness after fabrication and final etch. The supports were to be "hard-sized" in the driver inside diameter.

Very early in the program, measurements of the elastic strain in the sized support assembly showed that the "spring-back" was just sufficient to assure that the supports would maintain contact with the driver inside diameter at operating temperatures. Hydraulic testing at temperature showed that, as the annulus increased because of the unfavorable differences in temperature and thermal coefficient of expansion between driver and target, the "hard-size" feature of the supports was diminished and the supports became springs with a very low

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load-to-deflection characteristic. The target was free to vibrate in the coolant stream, and failures occurred by fretting of the driver clad or by fatigue of the supports. Attempts to obtain higher spring stiffness by increasing strip thickness were not successful. The thicker strip could not be formed, without cracking, to the "buggy-spring" shape with the coproduct annulus dimensions.

Forming the thicker strip to a height in excess of annulus dimensions, "interference fit," was successful; but spring stiffness was not yet sufficient to avoid failures of the type mentioned. Doubling the number of supports per target and using a nominal diametral interference of 0.050 in. gave more satisfactory performance in flow loop tests and in-reactor, but the support system was completely redesigned to employ two rigid supports and one (or two) interference-fit supports at each end of the target. The prototype design, using two of the "buggy spring" supports in axial alignment and two inelastic supports spaced radially 120° at each end of the target, performed satisfactorily both in test loop charges and in-reactor. The two buggy-spring supports were therefore replaced by a single spring support (See Figure 2).

The new spring support ("W" spring) was developed by optimizing the required compromise between springback, spring constant, and load at annulus.⁽¹³⁾ Spring and rigid support details are shown in Figures 12 and 13. The spring design was less demanding of material formability than the "buggy-springs" and could be formed by commercial fabricators. Zircaloy strip was furnished to component manufacturers in the interest of insuring consistent spring characteristics.

The rigid target supports were not material sensitive: providing corrosion resistance was adequate. These supports were produced from commercial sources of plate or wire.

FIGURE 12. "W" Spring (as formed)

FIGURE 13. Rigid Support (As Formed)

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FABRICATION PROCESSES

CERAMIC (SINTERED PRODUCT) PROCESS⁽¹⁴⁾

The sintering process was established around two basic criteria:

- The process had to produce a target core with physical characteristics capable of withstanding finishing operations and reactor processing.
- The process had to produce a consistent density in the product to maintain control of the Li^6 isotope level per unit of core volume.

The ceramic process was, therefore, directed primarily at achieving a consistent sintered density.

The pellet fabrication process was originally developed by BNW.^(15,16) This development covered the processing of eight LiAlO_2 powder batches, where each batch was an individual LiAlO_2 conversion kiln load. Because these batches had consistent fabrication characteristics, it was assumed that they were typical of powder to be received on a continuing basis. Based on the capability experienced with these powders, a sintered density requirement of $78 \pm 5\%$ of theoretical density (2.62 g/cm^3) was specified for production of LiAlO_2 pellets for the demonstration program. This density range was found to produce mechanically stable pellets, and was considered easily attainable. Therefore, Li^6 content on input powders was specified to provide the desired Li^6 content in $78 \pm 5\%$ dense pellet production.

The powder materials used for process development did not prove totally representative of the feed material received during the program. Sizable quantities of powder were received which did not react consistently to the sintering process and

numerous problems with a low density product occurred during the first few months of production. The overall program time schedule did not permit more specific control of the alumina-carbonate reaction process for feed material uniformity. Therefore, development work was concentrated on the sintering process for maximum flexibility in processing powder with varying characteristics to the specified sintered density range. The evolution of the process as a result of this development work is illustrated in Table III.

TABLE III. *Process Evolution*

<u>Process Step</u>	<u>Original Process at Start of PT-NR-75</u>	<u>Final Process</u>
1. Receipt and Analysis of Powder	Chemical analyses only	Chemical & reactivity analyses
2. Characterize Powder	Not done originally	Pretest to determine green density and sinterability characteristics.
3. Condition Powder	600 $\pm$ 10 $^{\circ}$ C for 2 hr, minimum	600 $\pm$ 25 $^{\circ}$ C for 2 hr, minimum
4. Ball mill powder for particle size	7 $\pm$ 1/4 hr or given number of revolutions based on mill load size.	Mill to specified bulk density (particle size), no time requirement.
5. Blend Additions into Powder	1 wt% each of lithium stearate lubricant and stearic acid binder blended for 1 hr.	1 wt% lithium-stearate lubricant blended 15 min. 1 wt% DIW as binder blended in 45 min more.
6. Press Powder into Pellets	40,000 $\pm$ 1,000 psi compaction pressure	Same as originally
7. Bakeout Lubricant and Binder	400 $\pm$ 15 $^{\circ}$ C for 2 hr, minimum	600 $\pm$ 25 $^{\circ}$ C for 2 hr, minimum
8. Sinter Pellets	1325 $\pm$ 5 $^{\circ}$ C for 16 $\pm$ 1 hr	1325 $\pm$ 5 $^{\circ}$ C for 14 hr, minimum

[REDACTED]

A detailed review of the major process steps is included in the following discussions.

Receipt and Analysis of Powder

All powder used for the program was reacted using a rotary kiln. Each kiln load was termed a batch of powder. On receipt, each powder batch was sampled for the following analyses:

1) apparent density, 2) total lithium, 3) spectrographic semi-quantitative, and 4) fluorine. Each fifth batch underwent the following additional analyses: 5) isotopic Li^6 , 6) water, 7) carbon, and 8) X-ray diffraction. Analyses 2, 3, and 5 were performed to insure that the powder conformed to appropriate Engineering Specifications. The remaining analyses were performed to gather engineering data. Results of these analyses are detailed in other reports in this area. (17,18)

Characterization of Powder

Problems with feed powder variability were encountered early in the production startup phase. Initial engineering effort was directed at insuring that strict conformance to the established process was being maintained. Secondly, tests were performed in an attempt to determine why powder lots were different. (19,20) The above work, including a review of the reaction process, did not reveal the cause(s) of variability. As a result the decision was made to develop the fabrication process around the assumption that feed material variation would occur.

Process development efforts revealed that sintered density was a function of:

- average particle size
- green (as-pressed) density
- an accumulation of other factors which herein is called inherent sinterability.

Figures 14 through 16 demonstrate these relationships to sintered density. Figure 14 shows how sintered density increases

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with decreasing particle size for a given powder lot. The general relation shown in Figure 14 is considered typical of all powder lots; however, the location of the curve may be shifted up or down for other powder lots depending on the green density and inherent sinterability characteristics. Figure 15 is included to show how average particle size varies with apparent density. This curve is fairly constant for powders of similar particle geometry, composition, and structure; however, variations to this curve will be discussed in the ball milling section of this report. The apparent density measurement operation was much simpler to perform than subsieve sizing.

Figures 14 and 16 illustrate the effect of inherent sinterability. Both figures show that under identical conditions of average particle size and green density, different powder lots will sinter to different densities. This phenomenon was attributed to the LiAlO_2 production process and to the combinations and levels of impurity content in the powders. It is believed that the presence of some compounds during sintering provides reactions and intermolecular motion which aid the sintering process. The number of impurities normally present in LiAlO_2 makes difficult any determination of those elements which affect inherent sinterability. Therefore, in order to establish processing conditions, a sample of each powder lot was pretested to determine sinterability. The pellet fabrication process was then specified for particular powder lots to provide the sintered density desired.

The sinterability pretest establishes the sintered density a particular powder lot will develop from a given particle size and green density. Pretests were usually performed on as-received powder; however, the same results could be obtained

FIGURE 14. Apparent Density Versus Sintered Density
Powder Lots A-33 and A-61

FIGURE 15. Average Particle Size Using Fisher Subsieve Sizer Versus Apparent Density Using Scott Volumeter for High As-Received Apparent Density Powders

FIGURE 16. Green Pellet Density Versus Sintered Pellet Density Powder Lot A-193 and PT-66 Powder

from ball milled powder. Given a pretest bulk density, green density, and resultant sintered density, the powder was then rated as to sinterability and was assigned a set of process conditions for fabrication into pellets. The powder sinterability rating was calculated by the following formula:

$$S = (SD_0 - GD_0)(BD_0),$$

where S is the powder sinterability rating, SD_0 is the pretest sintered density, GD_0 is the pretest green density, and BD_0 is the pretest bulk density. The sintered and green density terms are percent of theoretical density (2.62 g/cm^3), and the bulk density is grams per cubic inch. This combination of units provides a convenient range of sinterabilities for comparison. For example, of the powder lots so characterized, Powder Lot A-108 had the lowest sinterability rating of 26, and Powder Lot A-70 had the highest sinterability rating of 190.

Given the pretest information, it is possible to calculate the sintered density which will be developed from a given average particle size, as indicated by bulk density. Referring to Figure 14, it can be seen that the sintered density versus apparent density curves are nearly linear. The green density versus apparent density curve of Figure 17 is of the same general shape with lower slope. A curve (Figure 18) of sintered density minus green density versus apparent density is obtained by subtracting the green density curve from the sintered density curve. The curves in Figure 18 are most nearly linear, and the assumption of linearity is used to predict the sintered density obtainable from a given powder apparent density. The accuracy of such prediction is considered to be $\pm 1\%$ of theoretical density.

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FIGURE 17. Green Pellet Density Versus Powder Apparent Density
for Powder Lot A-193

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FIGURE 18. Powder Apparent Density Versus Adjusted Density for Powder Lots A-33 and A-61

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The final particle size chosen for the processing of a given powder lot depends on many considerations, including:

- a) The time required for particle size reduction at ball milling
- b) The pellet pressing production rate obtainable, and the press die-set wear rate, from a given powder particle size
- c) The desired sintered density as dictated by Li^6 control procedures
- d) The anticipated effects of the process used on sintered pellet quality, particularly as to effects on pellet machinability.

Consideration of items a, b, and d all tend towards larger particle sizes. Less milling time is required for larger particle sizes; pellets can be pressed faster with larger particle sizes; and, generally, pellets produced from larger particle sizes had better machinability characteristics. These considerations must be compromised with item c, since sintered density varies inversely with particle size. Once a particle size is selected for a powder lot, the sintered density which can be obtained is calculated. If the calculated density is below requirements, steps are taken to provide the incremental density necessary.

The incremental density can be provided for by either or both of two means: 1) increasing green pellet density, and 2) use of sintering agents. Green density increases were found to be feasible by the means explained below:

- Precompaction--as much as 2% density increase was obtained by precompacting powder at 40,000 psi and crushing for repressing.
 - Increased pressing pressure--as much as 0.5% density increase was obtained by pressing at 45,000 psi with existing die-set design (40,000 psi normal). The
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potential exists for a 1.5% density increase by pressing at a maximum of 60,000 psi, with a modified die-set design.

- Melting the stearic acid binder--green density increases from 3 to 6% were obtained by melting the binder in the powder prior to pressing. Large gains in green pellet strength were another benefit.
- Replacing stearic acid binder with dionized water (DIW)--an estimated 3% increase of theoretical density was obtained by substituting DIW for stearic acid. Large gains in green pellet strength were also achieved.

Sintered density increases obtained from the use of sintering agents were found to be much larger and more easily obtained than those resulting from increased green density. Five different additives were added to powder prior to the ball milling station to determine their effects on sinterability. Typical results of tests employing 1 wt% of each additive are presented below in Table IV. As indicated from control powder results, the test powder lot was one of low sinterability.

TABLE IV. *Additive Effects on Sinter Density*

Additive	Green Density, %TD	Sintered Density, %TD
Calcium Carbonate (CaCO_3)	68	77
Potassium Carbonate (K_2CO_3)	68	66.5
Lithium Silicate (Li_2SiO_3)	67.5	72
Sodium Silicate (Na_2SiO_3)	67	70
Magnesium Oxide (MgO)	68	72
Control (normal without additives) LiAlO_2 Powder	68	72

[REDACTED]

The significant increase in sintered density obtained by the use of CaCO_3 prompted further development work with this additive. Figure 19 shows typical sintered density variation with CaCO_3 concentration. The slope of the curve varies with powder lots; i.e., to obtain a 4 to 5% increase in sintered density may require CaCO_3 additions of 5/16 to 1 1/4 wt%.

The effect of CaCO_3 on sintered density is believed due to intermolecular motion created during decomposition of the CaCO_3 ($\approx 900^\circ\text{C}$) to the oxide (CaO) followed by several possible complexes of the oxide with available alumina and lithium-aluminate.

Powder Conditioning

As-received powder was first passed through a set of crossed grate magnets to remove tramp iron picked up in manufacture. The powder was then loaded into conditioning containers to a maximum depth of 3 in. for furnace heating at 600°C for 3 hr, minimum. The conditioning process step was originally intended to increase powder sinterability and reduce moisture content. It is now believed that conditioning was of value only in reducing overall moisture content, burn-off of some impurities, and improving reaction to ball milling. Conditioning effects on sintering can be detrimental if temperatures are allowed to approach the α - γ transition temperature of the LiAlO_2 since γ - LiAlO_2 is less active during sintering. The continued use of conditioning is questionable and tests to evaluate elimination of this process step are advised with sustained operations.

Particle Sizing by Ball Milling

The ball milling process was one in which the LiAlO_2 powder was tumbled with grinding media in large closed jars. The

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FIGURE 19. Amount CaCO_3 Addition Versus Sintered Density Powder Lot A-41

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grinding media used was high density alumina ceramic of various configurations, and the jars were porcelain lined. Particle size is reduced by the crushing action of the media on the powder particles. The ball milling of LiAlO_2 was along established industry recommendations for ball milling.⁽²¹⁾ Ball milling results in reduced average particle size as well as a modified particle size distribution, i.e., a modified distribution of particle sizes by weight percent of the whole. The rate of particle size reduction was found to depend on:

- a) The percent of mill volume charged
- b) The type of grinding media
- c) Mill jar rpm
- d) The particular powder lot
- e) The initial particle size.

Conditioned powder was ball milled to reduce average particle size. The smaller particle size was necessary to attain the desired sintered density in that pellet green density increased directly with reduced particle size, and sintered density increased directly with green density (see Figures 14 and 16).

The original process development called for control of sintered density by milling only a portion of the powder as required to obtain a given sintered density. The disadvantage of this procedure was that milling different portions of the various powder lots produced powders of varying particle size distributions. Particle size distribution and average particle size would therefore have to be controlled together to attain desired sintered densities from powder lots of differing sinterability characteristics. Considering these control complications, the ball milling process evolved to one of milling 100% of the powder for a constant time. This process eliminated variable particle size distributions between powder lots. The milling

time was established at 7 hr, which generally provided the minimum particle size for most lots.

Milling for 7 hr proved to be undesirable from the production time standpoint and the variable average particle sizes obtained. During production of start-up material, the average particle size obtained for various powder lots under constant milling conditions varied from 2.35 to 13 μ . This particle size variation produced 3 to 4% variation in sintered density and caused considerable variation in other fabrication characteristics, i.e., grinding, sawing, etc.

The development of ball milling as used for the demonstration program was initiated from tests to determine ball milling times for various apparent densities (Figure 20). Figure 20 clearly illustrates that powder lots differ in particle size reduction rate and the time required for a particular lot to reach a given particle size is dependent on initial particle size. Based on this analysis, particle size was specified after characterization of each powder lot, and milling was performed until the desired particle size was reached. Particle sizes generally selected ranged from 4 to 10 μ . The most frequently used size was 10 μ which requires from 1/2 to 2 1/2 hr milling time, depending on the powder lot. This milling process did not produce any significant variation in sintered density.

Addition Blending

After ball milling, those material additions required for pellet pressing were blended into the powder. Calcium carbonate (CaCO_3) as a sintering agent, may also be added at this station; however, CaCO_3 was normally added during ball milling to decrease particle size and to insure uniform distribution throughout the powder. CaCO_3 added prior to milling is more

FIGURE 20. Apparent Density Versus Ball Mill Time for Powder Lots A-33, A-51, and A-61

effective, per given weight of addition, than when added during blending. Those additions required for pellet pressing were lithium-stearate ($\text{LiC}_{18}\text{H}_{35}\text{O}_2$), which was used as a die lubricant; stearic acid [$\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{H}$], which was originally used as a binder; and dionized water (DIW), which was developed as an alternate to stearic acid.

The lithium stearate serves to 1) aid (through lubrication) the flow of powder particles during the pressing operation, 2) minimize the buildup of internal stresses which result in cracked green pellets, 3) reduce die-set wear, and 4) reduce pellet ejection forces at completion of the pressing operation. The function of stearic acid or DIW additions was to increase green strength in pressed pellets. Blending practice has been to load the LiAlO_2 powder plus all additions into the blender without specific metering of one component into the other. A blending time of 1 hr has proven sufficient for thorough mixing. The lithium stearate must be blended in the powder for at least 15 min prior to addition of the DIW. Forty-five minutes additional blending time is then required. This procedure was required because the DIW wet the powder, and adequate mixing of the lithium stearate was not possible.

Pellet Pressing

The pellet pressing operation compacted the blended LiAlO_2 powder into the approximate size and shape desired in the final LiAlO_2 product. The sintered density in the finished pellets was directly dependent on the green density developed from pressing. Green (as-pressed) pellets were produced by compacting blended powder within a die-set at 40,000 psi pressure. This pressure generally developed a green density of 60 to 70% of theoretical density.

Pellet pressing was regulated to meet the following requirements:

- Green pellets had to be of sufficient green strength to withstand handling without chipping or cracking.
- The desired green density had to be consistently obtained.
- Pellets had to be produced at the fastest rate possible while maintaining pellet quality.

Pellet green strength was a function of the powder average particle size, compaction pressure, powder moisture content, cycle dwell time at high pressure, and the speed of pellet ejection from the die. A precise measure of green strength was not developed during the Coproduct Program; however, major differences in green strength were readily apparent. Subjectively, green strength increases directly with average powder particle size and powder moisture content (DIW content). Development of maximum green strength was compromised by the fact that die wear also increases with increasing average particle size, and pellet ejection forces increase with increasing powder moisture content. Die wear rate was usually judged of secondary importance compared to pellet quality and pellet production rates.

The optimum moisture level in the powder was at that point where the powder slowly "creaks" and "groans" while at high pressure. No noise was discernible when insufficient water was present. A loud "screech" occurred when the moisture level was excessive. These less than qualitative measurements emphasize the "art" approach to pellet pressing. In most cases, 1 wt% of DIW was considered optimum for powder of a ball milled apparent density of 13 to 14 g/in.³. The green strength provided by DIW was directly related to the quantity of DIW per unit of particle surface area. Powders of smaller average

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particle size (greater total surface area) required more DIW for optimum green strength than did powders of large average particle size (smaller total surface area).

Green strength increased with increased cycle dwell time at high pressure (40,000 psi) up to a maximum of 5 to 6 sec of dwell. The powder compaction occurred very rapidly in the first second of high pressure and, thereafter, proceeded more slowly. This phenomenon could be audibly detected during the pressing cycle. The "creaking" sound occurred rapidly at first and then diminished to silence. The "creaking" was indicative of the powder particles building up compressive stress to a certain level at which the powder compacted. This buildup and release of stress continued until the pressure was no longer able to overcome the compressive stress within the powder. To insure acceptable green strength, pellets were pressed with a two second minimum cycle dwell at high pressure. Under optimum conditions, acceptable pellets were produced with as low as one-half second high pressure dwell time. Manipulation of high pressure dwell time was one technique commonly used to control green strength, i.e., a way to increase the green strength without adding DIW to the powder was to increase the high pressure dwell time.

Pellet green strength varied inversely with the ejection speed from the die. Green pellets incur compressive strain at the high pressure portion of the pressing cycle. As pressure is released, the compressive strain is released and the pellet expands. The rate of pellet expansion varies directly with the speed of pellet ejection, and pellet green strength decreases directly with the rate of pellet expansion. The acceptable speed of ejection was facilitated by using die bodies with an ejection taper. Standard pellet dies contained

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an ejection taper of 0.014 in. per inch of length along the last 1 to 1 1/4 in. of the die cavity. These dies allowed for a maximum acceptable ejection speed of approximately 1 in./sec which resulted in a 0.005 to 0.010 in. diametral expansion of the pellet. Expansion can be maintained less than 0.005 in. with slower ejection speeds; however, the ejection speed used in practice was the fastest speed which produces crackfree pellets of acceptable green strength. This speed varied between blender loads of powder as the average particle size and moisture content varied.

Pellet green density was held consistent throughout a powder lot by providing uniform preparation for pressing and by maintaining the die-set in good condition. With a given average particle size, particle size distribution, and blending additions, the powder will press to a green density which is dependent on the pressing pressure used. Pressing pressure can vary with the condition of the die and punch set; i.e., punches, that are galled or loaded up with powder or lithium stearate, require much of the applied pressure to overcome friction within the die-set. This loss of pressure to friction reduces the compaction pressure with resultant reductions in green density. Green density versus applied pressure for three typical powder lots is shown in Figure 21.

Excess lithium stearate and/or moisture content within the powder also had a reducing effect on green density. High moisture content resulted in increased green strength, to that point where the force required to overcome shear strength of the powder boundary layer was significant compared to the force applied to the powder. The same phenomenon occurs with excessive lithium stearate. The force of friction at the boundary layer (die powder interface) liquefies the lithium stearate which produced a sticky pellet surface, and also developed a very high green surface strength which increased required force for a given density.

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FIGURE 21. Green Density Versus Pressing Pressure for Three Powder Lots

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Pellet pressing rate was dependent on green strength and average particle size of the powder. When the powder was milled to an apparent density of 13 to 13.5 g/in³ (corresponds to average particle size of 9 to 11 μ) or greater, pellet pressing rates were significantly increased, i.e., an average particle size of 3 to 4 μ established a maximum pellet pressing rate of 2 pellets/min versus 5 pellets/min obtained with an average particle size of 10 to 11 μ . The relationship between pellet pressing rate and average particle size was a function of press tooling design. The tolerance gap between the punch and die cavity allowed powder leakage around the punch during pressing. This leakage was a function of particle size and pressure losses due to associated friction vary accordingly.

The pellet size produced during a major portion of the demonstration program was 2.75 in. long x 1.165 in. OD. Pellets were pressed in lengths up to 3 1/2 in.; however, the higher length to diameter ratios did not prove practical for production purposes. The green pellet diameter selected was based on typical shrinkage values during sintering. The die bore size was selected to provide sintered diameters 0.025 to 0.030 in. larger than the finished outside diameter to allow for centerless grinding.

Bakeout Lubricant and Binder

The bakeout process provided for the controlled decomposition and removal of moisture and organics from green pellets. The evolution of gases occurs at a rate which is dependent on the temperature and quantity of moisture and organics in the pellets. This rate must be controlled to prevent bursting of green pellets. Green pellets were baked out prior to sintering

since the sintering temperature would cause rapid evolution of gases with resultant pellet decomposition. Table V lists data pertinent to the bakeout of water (H_2O), lithium stearate ($\text{LiC}_{18}\text{H}_{36}\text{O}_2$), and stearic acid [$\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{G}$]. The additives are listed in the relative order of bakeout initiation and completion.

TABLE V. Binder and Lubricant Melting and Boiling Temperature

<u>Compound</u>	<u>Melting Point, °C</u>	<u>Boiling Point, °C</u>
H_2O	0	100
$[\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{H}]$	80	380
$\text{LiC}_{18}\text{H}_{36}\text{O}_2$	220	--

Lithium stearate and stearic acid turn brown upon melting. Figure 22 shows the cross sections of pellets baked for various times at 400 °C. The dark areas indicate the depth at which melting of either organic occurred. Visual examination indicates that the majority of the stearic acid was baked out after 10 min of heating; however, between 1 and 2 hr were required to drive off most of the lithium stearate. Using the visual examination approach illustrated in Figure 22, tests were performed which proved that a 600 °C bakeout temperature could be used on a shorter time cycle and thus eliminate thermal shock when transferring the pellets into sintering furnaces idling at 800 °C. Visual observation of pellet cross sections provided only a gross measure of additive removal. Residual carbon content analyses from each pellet shown in Figure 22 showed that approximately 90% additive removal occurred. The remaining additives were effectively removed in the sintering operation without any detrimental effects on the pellets.

Sintering

The sintering operation increased pellet density, pellet strength, and pellet surface quality. The sintered density of

5 10 15 30

Time, min

60 120 180 240

Time, min

Neg 0661890-1

FIGURE 22. 400 °C Bakeout--1 wt% Lithium Stearate
and 1 wt% Stearic Acid

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finished pellets directly affects the Li^6 content per unit of LiAlO_2 volume (also Li^6 content/unit of target volume). All pellet fabrication process steps prior to sintering were directed towards development of the desired sintered density.

Green LiAlO_2 pellets were sintered by heating to a temperature approaching the melting point of LiAlO_2 . At this temperature the LiAlO_2 particles are in more intimate contact, the effectiveness of surface tension reactions is increased, plasticity is increased, and there is the possibility that better mechanical interlocking is produced by the infiltration of a fluid network comprised of some compound(s) present which are in the liquid phase at sintering temperature. The effectiveness of the sintering process is believed enhanced by any reactions occurring which produce intermolecular motion. A typical sintered structure is shown in Figure 23. The rounded edges of void spaces contained in the sintered structure shown suggest the presence of a liquid matrix during sintering.

The sintering process was performed in air at 1325 and 1335 °C with times varying from 12 to 17 hr at temperature. Sintering was performed in batch type furnaces heated by silicon carbide globar elements. One of the major sintering problems was maintenance of a constant sintering temperature throughout the furnace heating zone. Although considerable care was taken to provide more globar current to the front and rear heating zones and insulation of all access openings, thermal gradients still existed in the furnace and a 3 to 5% sintered density variation often occurred in a furnace load.

A variety of sintered pellet qualities were experienced during the Coproduct Program. Figure 24 illustrates the appearance of certain pellet types which are attributable to definite

Neg 566-6264-B

750X

FIGURE 23. LiAlO_2 Sintered Structure

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24a

24b

24c

Neg 0663160-2

FIGURE 24. LiAlO_2 Sintered Pellets

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causes. The barrel shaped pellets of Figure 24a can be the result of: 1) barrel shaped green pellets due to "bellied" die liner or 2) incomplete sintering. In the event a worn die liner was used, pellets will probably be of acceptable density. Straight green pellets which emerge barrel shaped from sintering have not received sufficient heating during sintering. Typically, pellet ends sinter first as they experience longer exposure to sintering temperature than do pellet centers. When barrel shaped pellets are produced from a sinter run, they can be further sintered by recycling. Figure 24b illustrates the appearance of normal pellets which have passed through the shape of those in Figure 24a, but have received sufficient exposure to sinter completely. Figure 24c shows the typical shape of pellets which have been densified by 30% or more during sintering. These particular pellets had a green density of 62% of theoretical and a sintered density of 85% of theoretical (a 37% increase). The "hour-glassed" pellets indicate a greater than normal amount of liquid phase present during sintering. This effect can be induced by using CaCO_3 or other agents which promote sintering.

Sintering has proven to be effective in volatilizing undesirable impurities in the LiAlO_2 . There is generally a 50% reduction in boron content and a 90% reduction in fluorine content during sintering. Unfortunately, lithium is also vaporized during sintering. The lithium loss has been measured at 2 to 4% of the original lithium content. This loss is accounted for in the procurement of powder and the adjustment of Li^6 content prior to powder processing. Lithium loss varies directly with exposure time to sintering temperature; therefore, increased loss must be accounted for when pellets are resintered or sintering times are changed.

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Implications for the Future

The LiAlO_2 fabrication process has two major shortcomings. One is that process development proceeded on an empirical basis, with a minimum amount of underlying development as to the causes of variable LiAlO_2 fabrication characteristics. The second shortcoming is that the process is engineering oriented, i.e., the process requires substantial engineering input as opposed to a standardized production process. These shortcomings are a direct result of limited time availability for process development work. The process could be continued as now defined, as successfully demonstrated by the Coproduct Program; however, certain changes would be desirable prior to application to a long-term production situation. A sustained manufacturing situation should include the following recommended actions:

- a. The LiAlO_2 preparation process (conversion from LiOH to LiAlO_2) should be investigated to provide procurement specifications to insure LiAlO_2 of uniform fabrication characteristics. The desired goal would be to receive powder which would consistently achieve a desired sintered density $\pm 1\%$ under constant processing conditions. Such powder would eliminate the present requirement for variable processing conditions, and would further benefit the Li^6 control problem by yielding a consistent sintered density.
- b. Process refinement or simplification is desirable so that production functions could assume those duties performed by Quality Control and Engineering for demonstration purposes.

ASSEMBLY (CLADDING) PROCESS

The assembly cladding process was an adaptation of the ceramic target for irradiation in the N-Reactor environment. This portion of the process was therefore directed at:

1) encapsulation of the ceramic pellets into aluminum for hydrogen isotope containment and postirradiation handling, and 2) assembly of aluminum clad assemblies into zircaloy cladding to provide required resistance to corrosion in high temperature pH-10 water. The assembly process was therefore directed primarily at physical integrity. A review of the major process steps is included in the following discussions.

Pellet Finishing Operations

Sintered pellets were checked for conformance to density specifications and then processed through three finishing operations including: 1) centerless belt grinding for outside diameter finishing, 2) end squaring by belt sanding to obtain a flat mating surface between pellets, and 3) abrasive sawing to pellet stack length for assembly into aluminum cladding components. These operations are shown in Figures 25, 26, and 27.

The major problem encountered in these operations was variable pellet density. Pellet reaction (material removal rate) to abrasive finishing equipment is affected primarily by density. Centerless grinding in particular, was extremely sensitive to this characteristic and grinder set up was checked and in most cases modified for each significant change in pellet density.

Ceramic Assembly into Aluminum Cladding

The most critical assembly problem in the various assembly operations was assuring that the moisture content of the ceramic was less than 150 ppm in the aluminum clad assembly. The pellet drying and aluminum canning process utilized was based on the

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Neg 670919-2
FIGURE 25. *Centerless Belt Grinding Outside Diameter Finishing*

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Neg 670919-3

FIGURE 26. *Belt Sanding End Squaring*

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FIGURE 27. Abrasive Saw to Stack Length

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following developed ceramic-moisture relationships:

- a. 500 to 600 °C is sufficient to drive all moisture from the LiAlO_2 regardless of initial moisture content, i.e., pellets soaked in water prior to drying did not exceed moisture levels typical of standard production pellets.
- b. Moisture adsorption by the LiAlO_2 will not occur above 500 °C.
- c. Dried LiAlO_2 adsorbs moisture at the rate of 1 ppm/min from a "dry" atmosphere (4 to 6 ppm level). This emphasizes the importance of controlled exposure time after cooling below 500 °C.
- d. The LiAlO_2 moisture adsorption rate is high initially, but the increase declines asymptotically as ambient conditions are approached. Maximum moisture content which can be reached is not known. The maximum content would not be limited by the atmosphere moisture level if unlimited exposure time was allowed.
- e. Different reaction batches of LiAlO_2 adsorb moisture at different rates. No correlation was found between other LiAlO_2 characteristics and the rate of moisture adsorption. Moisture adsorption rate is considered a function of the types and amounts of various chemical compounds in the LiAlO_2 .
- f. No correlation was found between pellet density and moisture content or rate of moisture adsorption.

The assembly process used is summarized in Table VI. This process, using the maximum exposure times, consistently produced canned LiAlO_2 moisture levels of 75 ppm or less. Normal production practice quite often reduced exposure times with resultant lower moisture levels. The average moisture content for all target assemblies produced during the demonstration program was below 50 ppm.

The glovebox assembly chamber referred to in Table VI is shown in Figure 28.

TABLE VI. Pellet Drying and Assembly Process

<u>Process Step</u>	<u>Exposure Time</u>	<u>Maximum Atmosphere Moisture Content</u>
a. Furnace Drying (650 to 850 °C)	2 hr minimum	Room Air
b. Pellet transfer (furnace to glovebox cooling chamber)	90 sec maximum	Room Air
c. Cool pellets in chamber	45 min maximum	100 ppm 1/2 hr after charging (argon)
d. Assemble into aluminum	90 min maximum	600 ppm (argon)
e. Glovebox in-process storage (capped-unwelded assemblies)	60 min maximum	600 ppm (argon)
f. Preweld in-process stor- age (capped-unwelded assemblies)	60 min maximum	Room Air

Two alternative methods of controlling pellet moisture content were investigated. Pellets were vacuum dried versus 800 °C furnace drying. Holding time in vacuum proved excessive to attain moisture levels below 150 ppm. Pellets were assembled at 750 °C into aluminum cans at 200 °C. This hot assembly approach was quite successful in holding moisture levels below 50 ppm. The hot assembly process offers the potential for reduced capital investment and lower operating maintenance is a sustained operating case.

Aluminum Assembly End Closure Welding

The Coproduct Target aluminum subassembly end closure weld is made with the tungsten inert gas process using an ac power supply.^(22,23) The ac power supply was chosen over dc because

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FIGURE 28. Glovebox Assembly Chamber

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ac does provide some degree of cleaning by cathodic etch during the welding process. Figure 29 shows a cross section of the aluminum end cap and welds. The step in the cap outside diameter allows the welding arc to provide a cleaning action between the faying surfaces. The Number 55 drill hole in the center of the cap is a venthole to allow escape of entrapped atmosphere as the cap heats during welding. This precludes blowout of the outer weld. The vent hole is sealed after the main closure is complete. Success of the vent hole closure process depends upon the speed with which the closure can be completed. The 0.25 in. center section is fused into a smoothly rounded dome. The step in the clad inside diameter provides positioning and thins the wall of the can to provide a thermally balanced weld joint.

Rotation speed of the aluminum subassembly was 6 to 7 rpm during welding of the outer weld joint. A semiautomatic programmer provides a dwell time before rotation starts after initiation of the weld cycle. During the dwell time the current is increased from zero to 150 A. After the 0.1 to 1.0 sec dwell time, rotation of the work piece starts and the current is decayed automatically to 95 A. The weld is completed at 95 A. At completion of the weld, the operator initiates the stop cycle which decays the current to approximately 20 A before the weld contactor is opened. Decay time is 120° of rotation maximum.

The work piece was not rotated during the vent hole weld. The electrode is positioned directly over the vent hole during welding. The current program provides current slope control to 150 A and decay slope to approximately 20 A. The seal weld was completed in 3 to 3.5 sec.

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Neg 566-6027-B

5X

Vent Hole

FIGURE 29. *Aluminum End Cap Welding*

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Aluminum Assembly Sizing

The aluminum assemblies were hydrostatically sized following end closure welding to reduce the assembly clearance between the aluminum clad inside diameter and the pellet outside diameter. The sizing operation was performed at autoclave conditions of 2500 + 50 psia and 150 + 50 - 10 °C for a period of 15 min minimum. Sizing to minimize clearances in the finished target assembly was necessary to eliminate potential zircaloy cladding collapse due to system pressure which could affect support performance.

Various mechanical sizing methods (die sizing, spin or roll forming, etc.) were evaluated versus the hydrostatic method. In general, most mechanical methods created problems in maintaining a low moisture level in the ceramic. In addition to processing advantages, the inherent ability of the autoclave to detect defect pellets and welds was a characteristic judged necessary for overall product quality.

Aluminum Assembly Finishing

The sized aluminum assemblies are straightened to 0.010 in. TIR maximum and belt centerless ground to eliminate surface irregularities and to size the outside diameter to fit the available zircaloy cladding components. This grinding operation essentially eliminates ovality and the assembly outside diameter variation was 0.003 in. maximum.

Aluminum assemblies were helium leak tested after grinding to assure that no pinholes or cracks were opened during the grinding process. The leak testing procedure is described in detail in a latter section.

Blackness Testing

To insure that aluminum assemblies containing significantly different levels of Li^6 did not become intermixed in the process,

[REDACTED]

a "blackness" tester was used to test 100% of the material processed against known standards. The "blackness" tester is essentially a device for measuring the relative neutron adsorption characteristic of each assembly.⁽²⁴⁾ An assembly was placed between a known neutron source and a neutron counter. The neutron count was then compared with that obtained from a known standard to establish the Li^6 level. The tester was proven capable of detecting the three enrichment levels of interest (spike, base, poison) and providing a qualitative measure of conformance to tolerance ranges. The tester only provided a general indication of enrichment level and could not be used to establish the actual Li^6 content of assemblies. The 305 Test Reactor was the only proven method for establishing quantitative Li^6 values and no definite correlation with the "blackness" tester was possible.

Aluminum-Zircaloy Assembly

The assembled target element is a mechanical assembly with no bond between the lithium-aluminate, aluminum, or zircaloy components. Assembly and thermal expansion clearances were provided in the target design to allow for differences between component in-reactor operating temperatures and thermal expansion. A basic mechanical design analysis of the target, under extreme-case conditions of assembled dimensions and in-reactor environment, indicated that the pellet-aluminum subassembly might expand enough longitudinally to incur more than 0.100 in. length interference with the zircaloy end caps.⁽¹⁷⁾

This condition could be experienced in a target located at a position of maximum heat generation (4.4 kW/ft) with minimum allowable longitudinal assembly and thermal expansion clearance (0.080 in.). In this case the longitudinal expansion of the aluminum was calculated to be 0.065 in. more than the zircaloy,

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and the lithium-aluminate 0.080 in. more than the aluminum; resulting in an equivalent forced interference of 0.145 in. between the aluminum and the zircaloy end caps. The equivalent force on the zircaloy end caps as caused by the internal interference, by the pressure increase due to reduction of assembly and expansion gaps by thermal expansion, by the pressure derived from gas generation within the ceramic, and by the external pressure of reactor coolant was calculated to be 372 psi. The resulting stressing of the zircaloy cladding would then be a factor of four less than the yield strength of the cladding. Irradiation experience to date has proven the target to be mechanically stable at design conditions.

The assembly clearance between the aluminum assembly outside diameter and the zircaloy cladding inside diameter was established at 0.006 ± 0.003 in. for standard operating procedures with a 0.012 in. maximum. Assembly clearances less than 0.006 in. have not been practical because of minor surface irregularities in the aluminum and/or zircaloy. Aluminum-zircaloy assembly is a selective assembly operation. The aluminum assembly outside diameter is dependent on both the zircaloy cladding inside diameter and the fabrication method used for producing the zircaloy cladding. The fabrication method is of importance in that the cladding inside diameter surface finish varies significantly between free-sink drawing and plug drawing. Assembly problems would be minimized with a standard fabrication route for all zircaloy cladding components.

The zircaloy end closures are made with the tungsten inert gas process using a dc power supply with helium as the inert gas. A typical weld cross section is shown in Figure 30. Helium was also introduced into the target assembly at this time to improve the overall reliability of helium leak testing after autoclaving.

Limit of Heat Affected Zone

Neg 566-6102-A

20X

FIGURE 30. *Typical Production Zircaloy
End Closure Weld*

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Support Welding

Self supports for the coproduct target element are projection welded to the zircaloy clad following end closure welding. The weld projection arrangement (multiple), illustrated in Figures 12 and 13, was developed for use with a spike welder powder supply to obtain maximum weld shear strength with minimum cladding penetration. Weld penetration was of particular concern since a bond could be developed between zircaloy and aluminum which was not compatible with postirradiation processing. The normal shear strength developed per weld tab was 600 pounds. A typical weld cross section is shown in Figure 31.

Autoclave Testing

The target assemblies were nitric-hydrofluoric etched (~ 0.001 in. removal per surface) and autoclaved to detect cladding penetrations and to assess the corrosion resistance of the zircaloy cladding. A superheated steam cycle was used: 400 °C at 1500 psi. The high temperature and pressure cycle was used also as a final test of the structural characteristics of the assembly in an environment in excess of reactor conditions, i.e., excessive diametral assembly clearances between the aluminum subassemblies and the zircaloy cladding were easily detected after autoclaving due to cladding collapse.

Leak Testing

Autoclaved targets were helium leak tested to assure clad and weld integrity. The targets were placed in an evacuation chamber, held at a low pressure for a minimum of 30 min, and the chamber was then backfilled with helium at a pressure of 1 to 2 psi for 15 min. The targets were then leak tested along with a leak test standard. The leak standard was processed through the complete cycle with an assembly lot of targets to assure against equipment malfunction.

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20X

Heat Affected Zone

FIGURE 31. *Typical Production Multiple
Projection Support Weld*

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DRIVER-TARGET ASSEMBLY

Assembly of the target element into the driver fuel was performed consistent with two major criteria:

- The annuli between the target outside diameter and the driver fuel inside diameter had to be within established tolerances.
- The "W" support spring characteristics had to be assured and maintained within the limits required to prevent breaking of contact between any of the support components and the driver cladding under the action of the maximum hydrodynamic forces expected during residence in the reactor.

Maintenance of an acceptable annulus variation was dependent on a number of inter-related variables including: target outside diameter, solid support height, driver inside diameter, and target cladding deflection due to the spring load in the support weld areas. Therefore, the assembly process utilized required measurement of the target outside diameter in the support location and grinding of the solid supports to a height compatible with specific driver fuels grouped by inside diameter.

The "W" spring force required to assure target stability during reactor service was established at 30 lb. The support system utilized called for cold assembly in the plastic range with a resultant loss of developed spring load due to stress relaxation at operating temperature. Stress relaxation of the Mark II "W" springs is generally as shown in Figure 32. To insure against significant changes in stress relaxation characteristics, sample springs were tested from each lot of supports, where lots are defined by strip rolling process lots or feed material. In addition, each assembly is tested cold for developed spring load following assembly. This testing

is performed by loading the target element against the "W" spring with a fixture designed for this purpose with empirically defined limits for developed load-deflection curves. (25,26,27)

The fuel assemblies were then shipped with the "W" springs in the top-center position to eliminate the possibility of handling damage. No attempt was made to orient the spring location during irradiation.

FIGURE 32. "W" Spring Stress Relaxation

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APPENDIX A

TARGET ELEMENT IDENTIFICATION AND MATERIAL CONTROL SYSTEM

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APPENDIX ATARGET ELEMENT IDENTIFICATION AND MATERIAL CONTROL SYSTEM

Material in process was controlled by use of Manufacturing Lot Record formats. Samples of formats used are presented as Exhibits I, II, and III. The "A" Format (Exhibit I) was used to control lots of material until pellets were formed into stack units. The "C" Format (Exhibit II) was used to control lots of material from stack sawing to assembly into zircaloy cladding. The "E" Format (Exhibit III) was used to control lots of material from the zircaloy cladding operation to final assembly with driver fuel.

The material identification system was based on a powder lot numbering system, an aluminum subassembly numbering system, and a finished target numbering system. The three systems were cross referenced in the respective Lot Record Formats.

The powder lot number was of the Form: A-123-123-78. All characters in a routine production lot were numerical except the first. The first letter identified the isotope level. The first set of numbers identified lithium-aluminate powder lot. The second set of numbers identified the sintering run within a powder lot. The third set of numbers was the sintered density attained as percent of theoretical. Significant deviations from the operating procedures in any of the powder processing operations required notation on the manufacturing record card. The powder affected by a deviation from procedures was coded with an alpha suffix on the appropriate set of numbers. The portion of a powder lot which was processed outside the operating procedures or was otherwise suspect for any reason, therefore, became a new lot

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starting at that point where the deviation occurred. This system minimized the problem of holding large powder lots for evaluation due to nonstandard processing of small portion of a lot.

The aluminum subassemblies were numbered sequentially with an alpha-numeric code of the following Form: AH 1 → AH-9999. The first alpha character identified the isotope level. A, C, D, and E were available for use on base material and S and P for spike and poison material respectively. The second alpha character (H, K, M, N, R, T, and U) was used only to minimize the number of characters used. The aluminum subassembly identification was referenced to a specific powder lot on the "C" Format.

The zircaloy clad target elements were numbered sequentially with an alph-numeric code of the following Form: A-1 → A-999999. The alpha character identified the isotope level. Letters for use were identical to those used on aluminum subassemblies. Short target elements (standard length = 26.440 in.) for use with reworked driver fuel elements were further identified by an L or N following the number series for 24.740 and 21.820 in. lengths respectively, i.e., A-267-3L was a base level 24.740 in. target element. The zircaloy clad element was referenced to the two aluminum subassemblies contained on the "E" Format.

EXHIBIT IA. FORMAT

Date

Lot#

LiAlO₂

LOT RECORD Oper Date Comments

1. Number of pellets _____			
2. Isotope content _____ (A,P,S, - Base Poison Spike)			
3. Cond. furnace # _____			
Temp. _____ Time In _____			
Temp. _____ Time Out _____			
4. Pebble mill: _____ Hours _____			
5. Blender _____ % steric acid, _____ % Li stearate.			
Other additives _____			
6. Pellet press _____ psi [] Manual, _____ [] Automatic _____			
7. Binder Bakeout Furnace _____			
Temp. _____ Time-In _____			
Temp. _____ Time-Out _____			
8. Sintering furnace# _____			
Time-In _____			
Time furnace reaches 1325 °C _____			
Time furnace is turned down _____			
9. Inspection density: _____ % theoretical _____			
10. Inspection sorting waiver diam _____, reject diam _____ TOTAL _____			
No. to be Waived _____			
Rejects _____			

(After fill-in)

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EXHIBIT IIC. FORMATALUMINUM SUBASSEMBLY FABRICATION

<u>RESTRICTED DATA</u>		<u>SUBASSEMBLY LOT #</u>			
This document contains restricted data as defined in the Atomic Energy Act of 1954. Its transmittal or the disclosure of its contents in any manner to an unauthorized person is prohibited.		Date			
		Al. Top Cap Lot #			
		Al. Bottom Cap Lot #			
		Pellet #			
		Al. Tube Lot #			
		<u>OPER.</u>	<u>DATE</u>	<u>COMMENTS</u>	
1. Centerless grind pellet(1.075±.002)					
2. Grinding inspection_____OD. Aug._____					
3. Stack length (12.640+/- .010)_____					
4. Drying furnace: Time in_____					
Time out_____(2 hr.min.)					
Temp._____(650°-850°C)					
5. Subassembly: Time enter.chamber_____					
Time into glove box_____					
Time capped (max.)_____					
Time leav.glove box_____					
6. Al. top cap weld time: Start_____					
Finish_____					
7. Moisture sample #_____					
8. Presizing inspection_____					
9. Sizing oper.:Time at 2500 psi & 150°C_____					
10. Warp check and straightening					
11. Sizing inspection					
12. Finish OD(grind or machine to size)					
13. Leak test					
14. Blackness test					
<u>Al Element#</u>	<u>Pellet Lot</u>	<u>Reject Category</u>	<u>Al Element#</u>	<u>Pellet Lot</u>	<u>Reject Category</u>
1			26		
2			27		
3			28		
4			29		
5			30		
6			31		
7			32		
8			33		
9			34		
10			35		
11			36		
12			37		
13			38		
14			39		
15			40		
16			41		
17			42		
18			43		
19			44		

(After fill-in)

~~CONFIDENTIAL~~

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EXHIBIT III

E. FORMAT

[REDACTED]
[REDACTED]
[REDACTED]
[REDACTED] d FINAL
[REDACTED] TARGET ASSEMBLY Date _____
[REDACTED] - (_____) Oper Date Comments

1.	Assembly into Zircaloy tube				
2.	Stamp identification on zirc end cap.				
3.	Insert end caps				
4.	Clean ends				
5.	Weld: Cap lot #				
6.	Inspection				
7.	305 Test ☐ Not applicable				
8.	Final Etch Target				
9.	Support Weld. Support Lot #				
10.	Flash Etch				
11.	Autoclave				
12.	Leak Test & Inspect				

Zirc Tube#	Aluminum		Reject	Disposition	Comments
	Head Lot	Tail Lot			
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					
21.					
22.					
23.					
24.					
25.					
26.					
27.					
28.					
29.					

UNCLASSIFIED

~~CONFIDENTIAL~~

~~RESTRICTED~~

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APPENDIX B

MAJOR EQUIPMENT ITEMS: TARGET ELEMENT FABRICATION PROCESS

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~~RESTRICTED~~

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APPENDIX B

MAJOR EQUIPMENT ITEMS: TARGET ELEMENT FABRICATION PROCESS

Ball Mills
27 Gallon Capacity
U.S. Stoneware

Blenders
60 pound capacity
Paul O. Abbe Inc.

Pellet Press - Automatic
Model HC-30
Hydromet Division, Olsen Mfg., Co.

Bakeout Furnace
Model L TE
Size 48 x 48 x 48
1250 °F Rating
Lindberg Hevi-Duty

Sintering Furnaces
2 each, Sargeant & Wilbur, Inc.
Size 12 in. x 16 in. x 96 in.
2500 °F Rating
1 each, Lindberg Model G-24
Size 20 in. x 24 in. x 48 in. - 2500 °F Rating

Drying Furnace
C.I. Hayes, Inc.
Size 24 in. x 24 in. x 48 in.
600 °F Rating

Centerless Belt Grinders - Pellets
4 inch belt
Engelberg Huller

Cutoff Saw
Model O
Pistorium

Glovebox, Controlled Atmosphere
Engineered Item

Aluminum End Closure Welder
Miller - ac
BWC-300-MAPA

Reactivity Tester
Engineered Item

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[REDACTED]

B-2

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Centerless Belt Grinder - Aluminum
8 Inch Belt
Engelberg-Hüller

Zircaloy End Closure Welder
Sciaky Power Supply
Model C-ZLR-Z, dc

Helium Leak Detector
Consolidated Electrodynamics Corp.

Support Welder
Model 10HF.2
Weldex

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[REDACTED]

**DATE
FILMED**

3 / 18 / 92