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TITLE

REVIEW - PROCESS DEVELOPMENT
COPRODUCT TARGET FABRICATION

AUTHOR

C. H. SHAW

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REVIEW - PROCESS DEVELOPMENT
COPRODUCT TARGET FABRICATION

C. H. SHAW
SPECIAL PRODUCTS ENGINEERING
N-REACTOR FUELS

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REVIEW - PROCESS DEVELOPMENT
COPRODUCT TARGET FABRICATION

The proposed coproduct target fabrication process is shown in Figure 1. A review of general background and process development(1) on each of the major process steps is included in the following discussions.

1. Lithium-Aluminate Powder

The standard process for producing lithium-aluminate is to react lithium carbonate with alumina in a kiln at about 750°C. The lithium carbonate is formed early in the process of separating lithium from its ores, and is the preferred salt for reacting with alumina to produce the lithium-aluminate.

The isotope ratio for the coproduct target element is considerably lower than that of natural lithium-aluminate. Therefore, the isotope ratio must be corrected by utilizing depleted material which is available from Oak Ridge as lithium hydroxide. The hydroxide is converted to carbonate by dissolving in water and treating with CO_2 and then reacted with alumina, as in the natural case. The resultant powder products (natural and depleted) are then dry blended to obtain the necessary lithium-6 per unit volume of finished product. As sufficient experimental data can be accumulated to assure that the lithia-alumina ratio is held reasonably constant in the reaction steps, it would be preferable to correct the isotope ratio in solution prior to conversion to carbonate.

Specifications on lithium-aluminate as detailed in RL-NRD-696, will have to be revised in line with the analytical capability of commercial sources. Mass spectrographic facilities will not be available at commercial sources routinely and it will therefore be necessary to work out some method of either: 1) pre-analyzing each batch of depleted lithium salts as available from Oak Ridge, or 2) analyze each batch of depleted lithium-aluminate in the vendor's plant and thus define the amounts of depleted and natural material to be blended. Assuming that a number of lots of lithium-aluminate can be produced from each lot of depleted salts, alternate 1) is preferable.

It is intended at this time to use 99.5 percent pure alumina. This represents an extra cost (five to ten percent over 99 percent pure alumina).

Experience with powder as proposed for use in the fabrication process has been limited to three shipments of natural material from Lithium Corporation of America. Lithium Corporation processing data on this powder is extremely limited, i.e., the reaction temperature is unknown other than the temperature normally used is 750°C. On-site analysis of as-received powders shows total carbon content variation from 2,000 ppm to 5,500 ppm, total lithium content variations from 8.2 w/o to 9.2 w/o, water content variations from 6.9 w/o to 0.3 w/o, and allotropic composition variations from 50 percent α - 50 percent γ (see Table I for detailed analysis results).

2. Powder Conditioning

All incoming powder is dried in air at 600°C for at least two hours. The powder is then cooled and stored in containers in a dry atmosphere. This particular step is necessary for several reasons. The obvious reason is to lower the overall moisture level in the as-received powder. The primary reason, however, is increased sinterability. Lithium Corporation powders sintered without this pre-conditioning step produced pellets with densities of 70 to 73 percent which were prone to chalk and/or crumble with handling. The same powder with the 600°C heating step sintered to 79 to 89 percent density depending on the allotropic ratio in the powder. The higher the α-phase content (30 percent more dense than the γ-phase), the higher the green pellet density with resultant higher sintered densities.

Allotropic ratio effects on sintered densities is an area of prime concern in the pre-conditioning step. Heating the powder to 1000°C for six hours has been found to reduce the α content of a sample from approximately 75 percent to less than ten percent. This heating was also beneficial in diminishing the residual lithium carbonate from, in this particular case, 2.3 w/o to 0.1 w/o. Heating the powder to 1000°C (recalcining) is not yet a proven benefit or necessity in the process. This recalcining step definitely reduces the sintered densities obtained which is undoubtedly due to the decreased amount of α phase during initial stages of the sintering operation. Lower density pellets are, if anything, more desirable from the fuel design standpoint, however, the fabrication assembly process now utilized does not admit to chalky or fragile pellets. Experience to date indicates that pellet densities of at least 75 percent are required for optimum assembly into the aluminum cans. Heating all powder materials to 1000°C might, in fact, improve the variability of sintered densities obtained, however, at this stage in the process development it is perhaps advisable to trade off lower pellet densities with little variation for higher pellet densities with some variation. In addition, recent experience with material provided by Lithium Corporation for the 20 tube test has reacted quite differently to the powder conditioning and pellet sintering process, i.e., the powder was heated to 600°C, but extremely low sintered densities were obtained.

In summary, a 600°C powder conditioning temperature was chosen versus the 1000°C recalcining temperature in the interest of insuring as-sintered pellet densities compatible with proposed assembly and sizing process steps.

3. Pebble Milling (Ball Mill)

Some percentage of all of the powder utilized will be pebble milled. This operation is performed in room air, however, this exposure is minimized since the pebble milling operation is performed in closed jars.

The amount of material milled and the milling time will be adjusted for incremental changes in sintered densities desired. It has been shown that as-sintered density increases with the percentage of material ball milled and the length of milling time. Again, the experience with various lots of material has varied considerably. From experience to date, it appears that during the early stages of production, until more detailed process control can be introduced into the powder vendor's plant, it will be necessary to adjust pellet density primarily by the amount of ball milling utilized. For example, the material now being processed for the 20 tube test is apparently going to require at least 200 percent more ball milling than material processed in the past.

4. Powder Blending

The powder is dry blended to blend the milled and unmilled powder and for binder addition. In general, 1.0 w/o stearic acid has been used as the binder for pellet fabrication. This amount of binder addition was arrived at by processing small batches of powder with 0.5, 1.0, 2.0, and 5.0 w/o binder additions. The criteria used for the amount of binder required was primarily pellet cracking following pressing.

The binder addition also acts as a die lubricant during the pellet pressing operation. In general, 1.0 w/o stearic acid has been adequate, however, recent shipments of powder have required an additional .5 w/o addition of lithium stearate to obtain adequate die lubrication (the stearate is a better die lubricant than stearic acid).

[redacted]

The major control points associated with this particular step in the process are time and binder particle size. In general, a blending time of two hours has been demonstrated to be sufficient for uniform distribution of binder and milled and unmilled powders. To insure against void formation by large agglomerates of binder, experience has shown that the binder should be passed through a 16-mesh screen prior to blending.

The blending operation as now planned would be performed in room atmosphere.

5. Pellet Pressing

Powder from the blending operation is placed directly into loading hoppers for the pellet presses. Measured amounts of powder are then loaded into the press dies and formed at approximately 40,000 psi. The green pellet length will be two to three inches with a density of 60 to 70 percent of theoretical. The green pellet diameter will be established to provide at least 20 to 30 mils of stock removal during centerless grinding from the minimum as-sintered diameter.

Figure 2 shows green pellet density versus pressure with associated as-sintered densities. It should be noted, however, that the data illustrated in Figure 2 was developed using powder produced by one particular method, i.e., the powder was dried at 600°C and ball milled for two hours. The relationship between green pellet density and as-sintered density is highly dependent on the ball milling time and also on as-received powder characteristics.

The green pellets will be inspected on a sample basis. Inspection will include primarily surface condition, dimensional checks, and density.

6. Binder Bakeout

The binder addition is removed by baking at 370°C. Experiments have shown that 30 minutes was required with the 0.5 and 1.0 w/o binder addition to eliminate binder from the pellets to the point that no more weight was lost with continued heating. One hour was required for the 2.0 w/o binder material to come to constant weight, and one and one-half hours were required to completely bake out the 5.0 w/o binder addition. With the above data, and the known characteristics of stearic acid (melts at 70°C and boils between 358 and 383°C), it was concluded that a bakeout cycle of two hours at 370°C would be sufficient for binder removal in the 1.0 to 2.0 w/o level. Pellets at room temperature should not be charged in a furnace operating at temperatures above 370°C since the binder must be removed slowly to preclude internal pressure buildup inside the pellets.

One of the major concerns with binder addition and the subsequent bakeout operation is the amount of residual carbon content in the pellets. There is some concern that all of the carbon is not removed from the pellets with a 370°C bakeout. However, following the bakeout all pellets are then transferred to the sintering furnace which is operating at an idling temperature between 800 and 1000°C. The present process flow therefore does not provide for pre-sinter bakeout temperatures above 400°C on the basis that the pellets will be raised in temperature from approximately 800°C over a four to six hour time cycle to 1325°C sintering temperature.

Experience to date on the three lots of material processed by Battelle-Northwest Laboratory shows carbon content variation in the as-sintered pellets from 19 ppm to 103 ppm.

7. Sintering

The sintering process now calls for a 12-hour cycle at 1325°C. Transfer of pellets to and from the sintering furnace will be performed at 800° to 1000°C (furnace idling temperature). Assuming a four to six hour temperature rise from idling temperature to 1325°C with approximately the same timing on the cooling cycle, the furnace turnaround time would be approximately 24 hours. Sintering is performed in air.

The actual time at 1325°C required for adequate pellet sintering has not been firmly established. Most of the experience to date has been with sintering times between 16 and 24 hours. Preliminary experience with sintered density vs. time at temperature is illustrated in Figure 3. As shown in Figure 3, pellet density is within one-half percent of the density attained in 24 hours with a 12-hour cycle. Further, at six hours pellet density is within two percent of that attained in 24 hours. It should be noted here, however, that this data was generated with one particular batch of input powder and there is no data at this time which would indicate that minor variations in process parameters would not significantly effect the shape of the curve shown in Figure 3. It is also of interest here that at time periods less than 12 hours the slope of the curve is changing rapidly and minor differences in time could cause significant changes in as-sintered density. In summary, a 12-hour cycle time (although this time has never been used) should produce stable pellets on a reproducible basis. To reduce the time cycle below 12 hours could definitely effect the reproducibility of material produced with very little savings in furnace turnaround time due to constant heat and cool cycles.

The sintering process will be directed toward producing pellets with approximately 80 percent density. The detailed process for sintering with varying input powder materials and processing has not been established. The process definitely has not been developed to the point at this time where reliable estimates of product can be made with known input materials. Process development work in this area will undoubtedly proceed well into the production period.

Sintered pellets will be inspected on a sampling basis for surface finish, cracks, porosity, dimensions, and density.

Sintered pellets will be transferred at 800 to 1000°C into a storage chamber. This chamber will provide for cooling in a dry inert atmosphere. Pellets will be stored in this atmosphere until required in subsequent processing steps.

8. Centerless Grind

All pellets are belt centerless ground to finished diameter following sintering. In general, the dimensional tolerances obtained with belt centerless grinding of pellets in the 78 to 85 percent dense range approach ± .002 inch. This operation is performed in room air.

Grinding is performed in room air primarily because of the associated cost in performing the grinding operation in a controlled atmosphere hood. The pellets are stored in a controlled atmosphere after sintering because the storage time in this case could approach 48 hours on a normal operating basis. Moisture pickup during grinding will definitely occur since the lithium-aluminate pellets act as a desiccant. Performance of this work in an atmosphere where moisture pickup can occur is justified primarily on the assumption that the pellets can be redried.

Immediately following the grinding step the pellets will be end squared by either grinding and/or sawing and the stack lengths required will be established by grinding and/or sawing. This operation is also performed in room air.

The pellet exposure time to room air and the associated moisture pickup problem will be minimized where possible. It is believed, however, that exposure to room air will probably be at least six hours. Data does not now exist on the rate of moisture pickup, however, the desiccant properties of the material leads to the assumption that equilibrium with ambient moisture is approached with any exposure exceeding one hour. Therefore, the centerless grinding and stack length operations in air admit to pellet exposure for a period of time which would allow significant pickup of moisture. Production scheduling to minimize exposure to periods less than one hour is not believed practical in this case.

9. Pellet Drying

All pellets are dried at 600 to 850°C for a minimum of four hours. The pellets are then transferred at a minimum of 600°C to a dry inert atmosphere hood. This process step is based on two pieces of data: 1) all moisture in a small powder sample may be eliminated in several hours at 350°C in a relatively dry atmosphere (bottle Argon)(2). 2) D. S. Knepple(3) found that two hours at 500 to 600°C in air was sufficient to remove all moisture from sintered aluminate pellets of 3/4 inch diameter. In both of the above references, the word "all" did not have specific ppm levels assigned, and neither case is necessarily pertinent to the specific process now proposed.

This particular process step and the times, temperatures, and atmospheres associated with it is undoubtedly the most important step in the process in minimizing the amount of moisture in target assemblies. If this process can be made efficient in removing all absorbed moisture from the pellets (open porosity moisture) then the target fabrication process as proposed would be satisfactory for the demonstration loads. The amount of moisture entrapped in the pellets (closed porosity) obviously cannot be removed by this drying step. Analysis of pellets dried for a two hour period at 600°C was performed by Battelle-Northwest Laboratory on pellets fabricated during the initial process development stages. (See Table II for detailed characteristics of Battelle-Northwest Laboratory produced pellets.) This analysis showed water content ranging from 71 to 483 ppm with most of the analyses in the 400 ppm range. There is still, of course, considerable doubt about the sampling and analysis technique used.

10. Pellet Assembly in Aluminum

This operation is performed entirely in a dry inert atmosphere. As indicated above, pellets are transferred from drying at 600°C into the assembly area. Aluminum canning components cleaned and prepared according to the standard process now utilized for aluminum clad fuel elements are heated to 400°C and transferred into the assembly area. These aluminum cans have the base end cap welded in place at the time of transfer into the dry inert atmosphere. The pellet assembly in aluminum cans is performed by a glove box arrangement. The pellets are loaded in the aluminum cans cold with an assembly gap of approximately six to eight mils. The second end cap is then placed on the aluminum can and the assembly transferred to an air lock attached and immediately adjacent to the inert gas glove box. This air lock arrangement is simply to minimize the time of exposure of the canned assembly to room air prior to welding the final aluminum end closure.

The inert gas to be used in the glove box will be either nitrogen or argon and will be passed through a drying step, i.e., bottle gas will not be used without drying.

[redacted]

11. Aluminum End Closure Welding

The aluminum end closure welding will be performed with an A.C. welding power supply. Minimum weld coverage shall be 50 percent of the aluminum clad thickness. The aluminum end cap now includes a small vent hole to prevent buildup of internal gas pressure in the assembly during welding with resultant weld blowouts. This vent hole is sealed by magnetic force resistant welding. An alternate process using the A.C. power source for sealing the vent could also be used.

The aluminum closure process will include end cap designs and will be scheduled to minimize the exposure of lithium-aluminate to air. The weld gas will be utilized such that helium will not be introduced into the aluminum assembly.

12. Aluminum Weld Inspection

The primary weld inspection step will be helium leak testing of all welds. This test will be performed by charging 100 plus aluminum assemblies in an evacuation chamber which are held at moderately low vacuum for a period of at least two hours followed by backfill of the chamber with helium. The elements are then removed from the chamber in small lots and placed in test chambers attached to a standard helium leak detector. This test is not considered as sensitive to small weld defects as a test which would include backfill of all aluminum assemblies prior to the final end closure welding followed by the standard leak check. However, the introduction of helium in 100 percent of the assemblies is not compatible with the extraction process.

X-ray examination and metallographic evaluation of aluminum welds will be performed on a sample basis to ensure adequate process control.

13. Aluminum Sizing

To minimize assembly gaps in the finished elements the aluminum cladding is sized to the pellet diameter. The present sizing process includes hydrostatic (autoclave) sizing at 1,500 to 2,000 psi at 250 to 350°C. The exact pressures and temperatures have not been established to date, but tests have been performed in most of the ranges available in existing autoclaves. In general, the process will be established on the basis of autoclave cycle time available with the minimum conditions required to remove all assembly gap.

The autoclave sizing process establishes a need for close tolerances on pellet end squaring and diameter. Tests performed to date have shown that only small deviations in end squareness can cause considerable warp in the as-sized assembly (up to 70 mils). Autoclave sizing also requires a machining or grinding operation on the aluminum assembly following sizing. This machining is required to reduce the diameter of the aluminum end caps to the diameter of the sized cladding.

Cold and hot die sizing of the aluminum assembly has been briefly investigated in the interest of minimizing the number of post-sinter operations on the pellets to improve tolerances. These processes are not compatible with the need for minimum moisture since the pellets are exposed to air for some period of time during and following the sizing operation and with sized aluminum cladding it is not possible to use high temperature drying operations for removal of moisture absorbed during the sizing operation. The possibility of cold die sizing in a controlled atmosphere hood still exists with further development work.

14. Nondestructive Testing - Lithium-6 Content

All aluminum assemblies will be tested using a neutron absorption test developed by Battelle-Northwest Laboratory.(4) This test will be used to insure that the given level of lithium-6 is correct (poison, base, spike). The present tester design is not capable of defining the absolute level of lithium-6, but will be able to distinguish between the three levels using known standards.

Samples of production material and nondestructive test standards will be tested for absolute values using the 305 Test Reactor.

15. Assembly of Aluminum in Zircaloy

The aluminum clad elements (see Figure 4) are assembled in zircaloy cladding with minimum diametral clearances (nominally six mils). The assembly will be performed dry without the use of lubricant. The assembly is performed from both ends of the zircaloy cladding, thus both zircaloy end caps are welded following assembly. The welding process is essentially that now used for coextruded fuels. Minimum weld coverage shall be 50 percent of the clad thickness.

The welding operation also includes a helium purge which will fill the void space inside the zircaloy cladding with helium for subsequent leak testing. The weld leak test is performed by placing the zircaloy clad assemblies in chambers connected to a helium leak detector.

16. Support Welding

Support welding will be performed with the spike welding power supply. The support projection will be multiple (four projections per tab). The support tabs shall be in contact with a target cladding after welding.

17. Autoclave

All target elements will be autoclaved at 1500 psi pressure and 400°C temperature for a period of 60 hours. The autoclave processing details are identical to those now used for standard coextruded fuels.

18. Final Assembly With Drivers

The final assembly process will vary as support concepts are modified to eliminate the potential of fretting corrosion failure. At present, the target supports will be: 1) 0.315 inch wide buggy springs which will be hard sized (six supports) or 2) standard width buggy springs modified for an interference fit with the driver I.D. using mild steel shoes on the supports (nine supports).

C H Shaw

Manager, Special Products Engineering
N-REACTOR FUELS

CH Shaw:mf

TABLE I

Characteristics of As-Received Lithium-Aluminate Powders(1)

(a) Batch Number	2	3a	3b
(b) Total Carbon - ppm	2068	5435	3743
Equivalent Carbonate-w/o	1.28	3.35	2.30
Lithium - w/o	9.2	8.3	8.2
Lithium - Isotopic - w/o			
a/o			
Water - w/o	6.9	0.3	
Average Particle Size-Microns	75	100	
Tyler Mesh	200	150	
(c) Approximate Allotropic	50%α	75%α	
Composition	50%γ	25%γ	
Element Lots	1,2,7 & 8	9 & 10	

(a) Each batch number refers to a separate shipment from the supplier. Letter after number refers to separate containers in a shipment.

(b) All carbon is assumed to be present as Li_2CO_3.

(c) Estimated from X-ray diffraction data.

TABLE II

Pellet Data Summary(1)

Sintering Run	First	Second	Third
Powder Batch	2	2	3a,3b
Element Lot Numbers	1,2	7,8	9,10
Density-Average-% of 2.62 gms/cc			
- Green	66.2	66.2	70.8
Sintered	79.6	79.0	88.6
After Grinding	79.6		
Diameter			
Green-Average	1.317	1.317	1.317
Sintered-Average	1.226	1.228	1.203
% Linear Shrinkage	6.9%	6.8%	8.7%
Sintered-Range	1.215" to 1/235"	1.222" to 1.238"	1.197" to 1.215"
Lithium Content - mmols/gm*	13.6	13.7	11.8
- mmols/cc*	28.4	28.2	27.4
w/o Li (assume natural isotopic distribution)	9.44	9.51	8.22
Measured Water Content - As	Lot 1 - 481	Lot 7 - 472	Lot 9 -
Loaded in AlCans - ppm	Lot 2 - 71	Lot 8 - 483	Lot 10 - 107
Carbon Content - ppm	51	103	19
Sulfur			300 ppm
Fluorine			< 10 ppm
Average Pore Size - Microns	3	3	5

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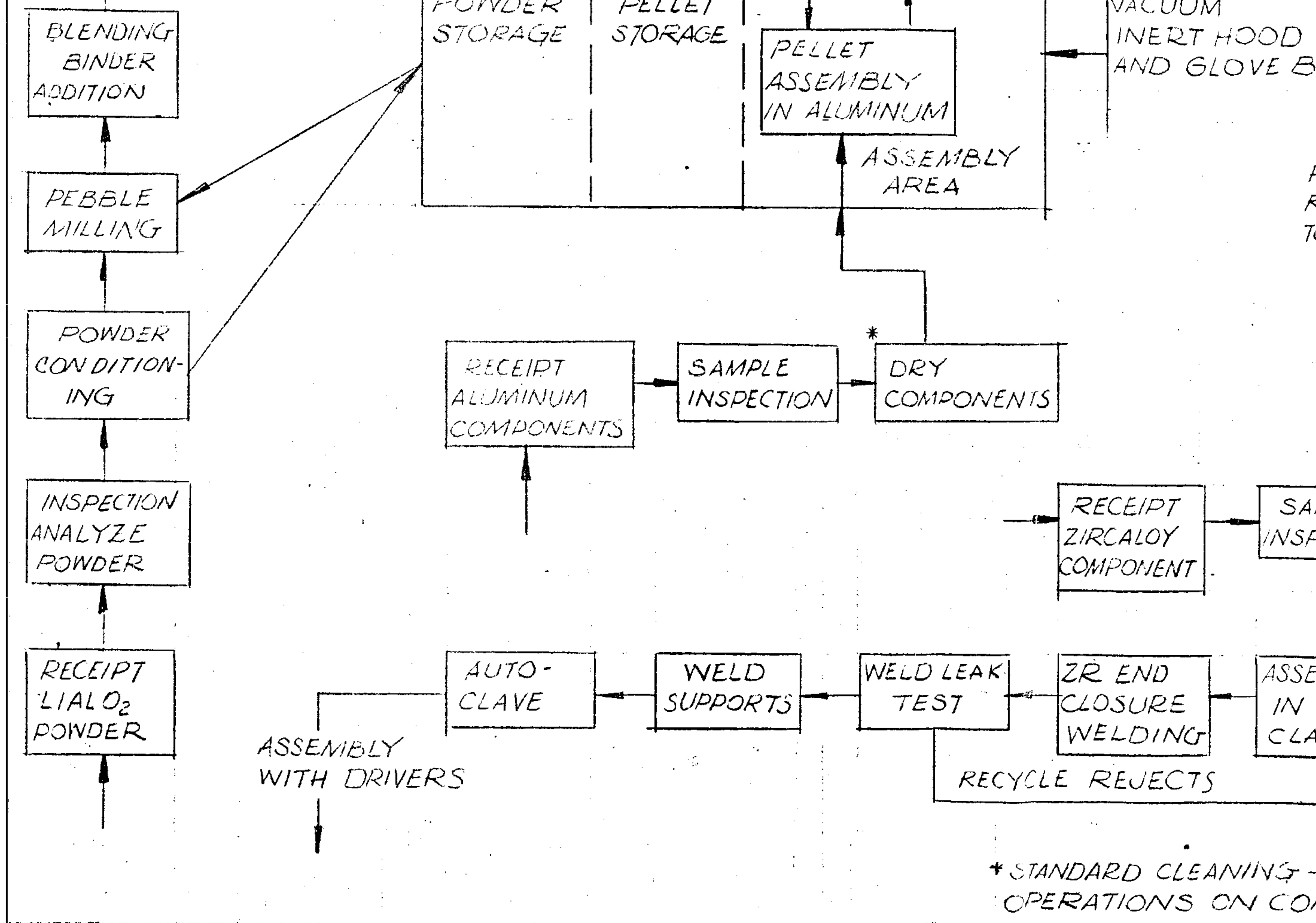
* 1 mmol Li = .00694 gms Li at normal isotopic distribution. Isotopic analyses have not been completed at the time of this report.



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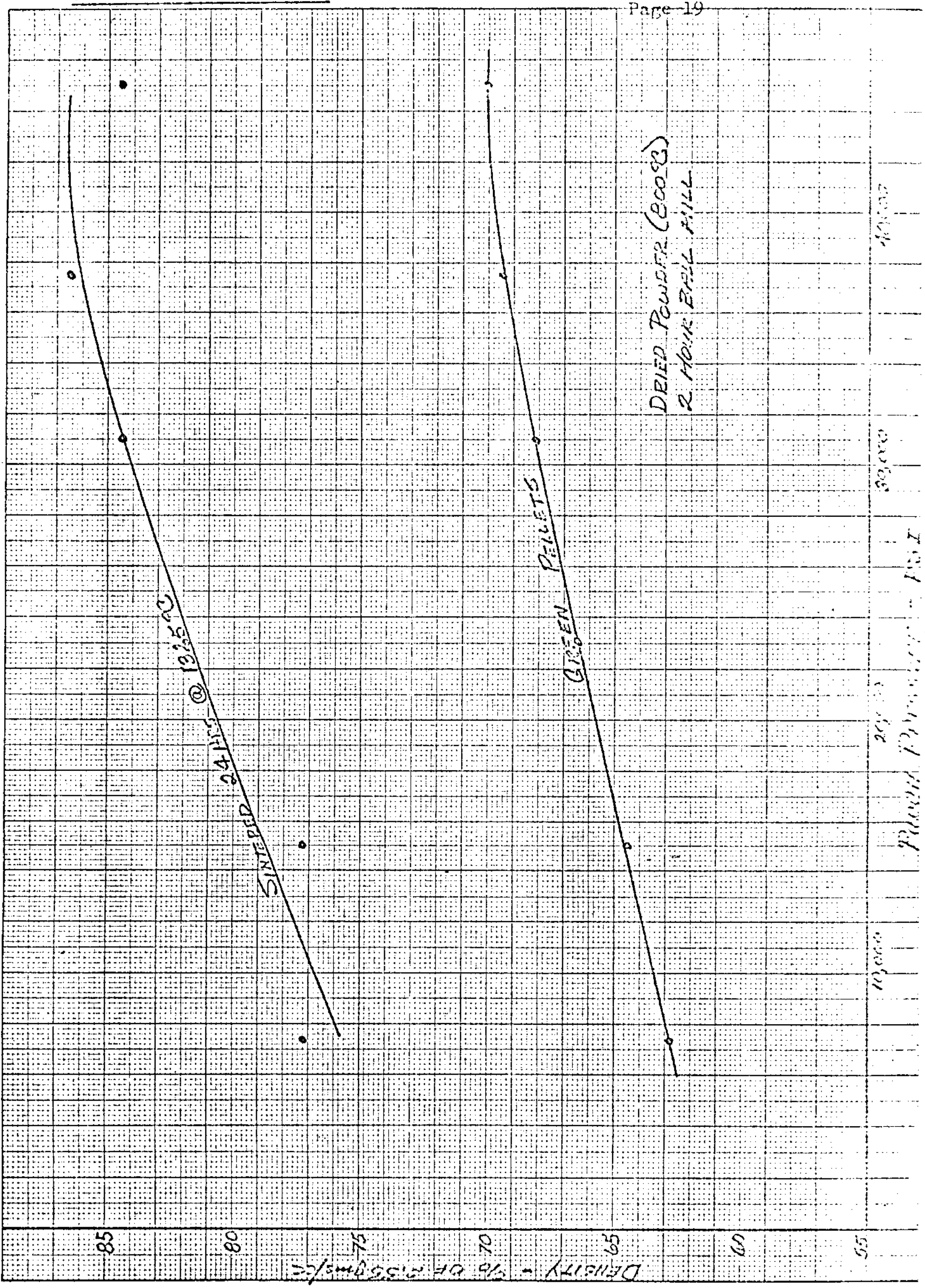
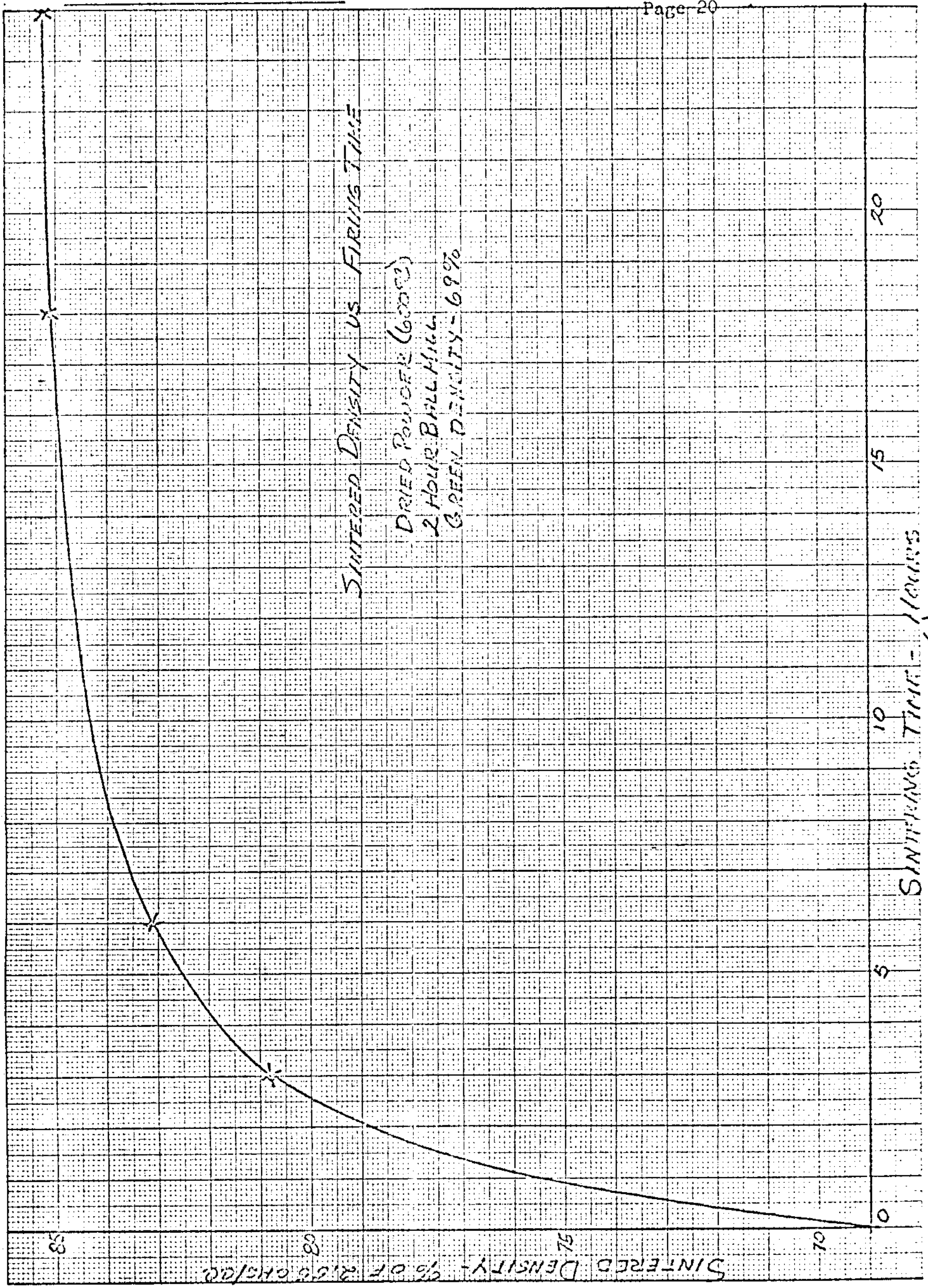
COPRODUCT TARGET FABRICATION

THIN-WALL ZIRCALOY CLADDING


DRIED POWDER (B00-B)
2 HOUR BALL MILL
SINTERED 24 HRS @ 1325°C
GREEN PELLETS
85
80
75
70
65
60
55
DENSITY - % OF 10.55 gms/cc
10,000
20,000
30,000
40,000
Pressing Pressure - PSI


SINTERED DENSITY VS FIRING TIME
DRIED POWDER (600°C)
2 HOUR BALL MILL
GREEN DENSITY - 69%
SINTERING TIME - HOURS
0
5
10
15
20
SINTERED DENSITY - % OF [illegible] GMS/CC
70
75
80
85
K&E 10 X 10 TO THE CENTIMETER 46 1510
10 X 25 CM.
MADE IN U.S.A.
KEUFFEL & ESSER CO.

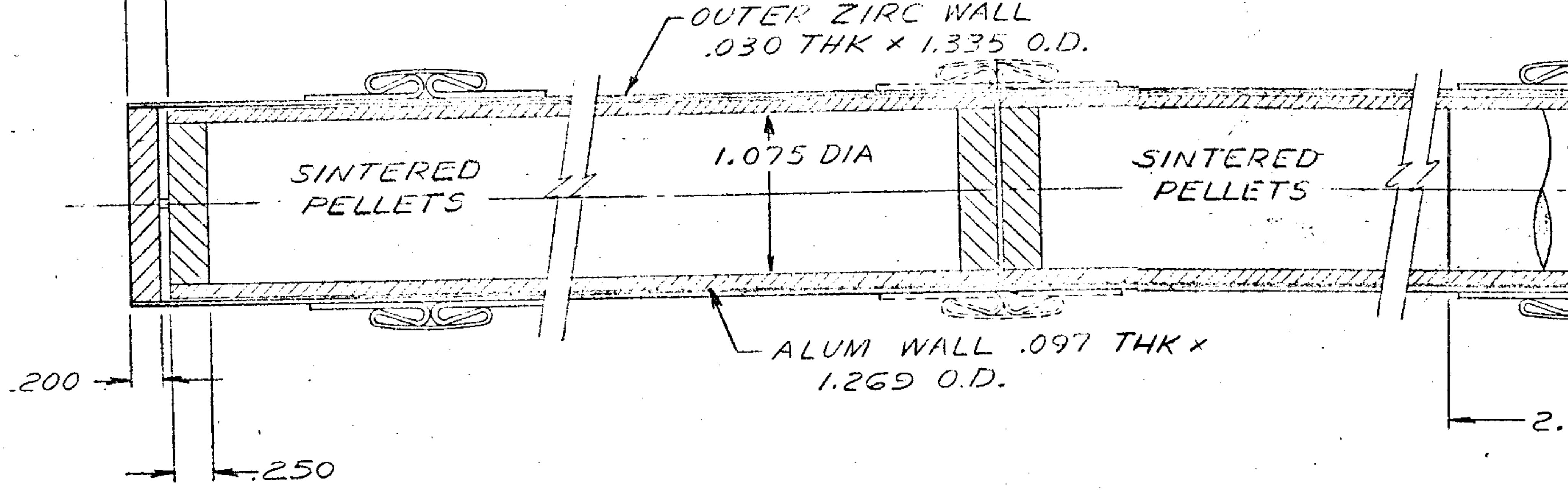


FIG. 4

CLASSIFICATION - NONE

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