[redacted] DECLASSIFIED

(CLASSIFICATION)

DOCUMENT NO.
RL-NRD-697 13
Hanford Code C-57

SERIES AND COPY NO.
[redacted]

DATE
March 30, 1967

GENERAL ELECTRIC

HANFORD ATOMIC PRODUCTS OPERATION - RICHLAND, WASHINGTON

[X] [redacted]

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.

[ ] OTHER OFFICIAL CLASSIFIED INFORMATION

THIS MATERIAL CONTAINS INFORMATION AFFECTING THE NATIONAL DEFENSE OF THE UNITED STATES WITHIN THE MEANING OF THE ESPIONAGE LAWS, TITLE 18, U.S.C., SECS. 793 AND 794, THE TRANSMISSION OR REVELATION OF WHICH IN ANY MANNER TO AN UNAUTHORIZED PERSON IS PROHIBITED BY LAW.

TITLE

MONTHLY ACTIVITY REPORT
SPECIAL PRODUCTS ENGINEERING
MARCH-1967

AUTHOR

C.H. Shaw

ISSUING FILE

BATTELLE-NORTHWEST
RECEIVED
APR 6 1967
CIRCULATING COPY
TECHNICAL INFO. SECTION

THIS DOCUMENT MUST NOT BE LEFT UNATTENDED OR WHERE AN UNAUTHORIZED PERSON MAY HAVE ACCESS TO IT. WHEN NOT IN USE IT MUST BE STORED IN AN APPROVED LOCKED REPOSITORY WITHIN AN APPROVED GUARDED AREA. WHILE IT IS YOUR POSSESSION AND UNTIL YOU HAVE OBTAINED A SIGNED RECEIPT FROM CLASSIFIED FILES IT IS YOUR RESPONSIBILITY TO KEEP IT AND ITS CONTENTS WITHIN THE LIMITS OF THIS PROJECT AND FROM ANY UNAUTHORIZED PERSON. ITS TRANSMITTAL TO, AND STORAGE AT YOUR PLACE OF RESIDENCE IS PROHIBITED. [redacted] NOT TO BE DUPLICATED. IF ADDITIONAL COPIES ARE REQUIRED, OBTAIN THEM FROM THE RELATED ISSUING FILE. ALL PERSONS READING THIS DOCUMENT ARE REQUESTED TO SIGN IN THE SPACE PROVIDED BELOW.

ROUTE TO:	PAYROLL NO.	LOCATION	FILES ROUTE DATE	SIGNATURE AND DATE
[redacted]				
			APR 6 1967	

Reviewed and Approved for
Public Release by the NSAT
[redacted] PNNL ADD
12.5.01 Date

54-3000-340 (3-57) AEC-GE RICHLAND, WASH.

[redacted] DECLASSIFIED (CLASSIFICATION) [redacted]

MONTHLY ACTIVITY REPORT SPECIAL PRODUCTS ENGINEERING MARCH 1967

C. H. Shaw, Manager
Special Products Engineering
N-Reactor Fuels Operation
Hanford Atomic Products Departme[redacted]

March 30, 1967

Distribution:

Atomic Energy Commission
1. O.W. Rathbun

Battelle-Northwest Laboratories
2. J.E. Minor/G.A. Last
3. J.C. Spanner/D.C. Worlton

General Electric Company

4. A.C. Callen
5. D.L. Cornell
6. D.W. Darsow
7. R.L. Dickeman
8. T.W. Evans
9. W.J. Ferguson/G.O. Amy
10. S.G. Forbes
11. A.E. Guay
12. J.E. Hansen
13. W.R. Kirk
14. H.P. Kraemer
15. E.F. Krautter
16. M.C. Leverett
17. M. Lewis
18. W.J. Morris
19. T.D. Naylor
20. A.R. Nichols
21. J.W. Nickolaus
22. W.W. Olson
23. J.W. Riches
24. C.H. Shaw
25. R.R. Studer
26. J.T. Stringer (DUN)
27. C.J. Volmer
28. D.H. Walker
29. W.K. Yale
30. 700 Area Files
31. Records Center

March 30, 1967

MONTHLY ACTIVITY REPORT
SPECIAL PRODUCTS ENGINEERING
MARCH 1967

PRODUCTION OPERATIONS

Fabrication of target elements proceeded on schedule for the month. Some delays in production occurred due to shortages of rigid supports and zircaloy tubing. These delays did not prove critical in that significant inventories of aluminum subassemblies were established and are available for immediate assembly into zircaloy.

Inventories of finished target elements have been increased over the past three months. All targets required for Charging Increment #4 were available prior to reactor use of Charging Increment #3.

ENGINEERING SUPPORT

Process areas requiring significant engineering time during the month were as follows:

Fabrication Characteristics $LiAlO_2$ Powder - J.E. Hansen

The reaction of input powder to the sintering process continues to change. The poison case $LiAlO_2$ powder processed in March was characterized by: 1) low green pellet density, 2) high particle size reduction rate during ball milling, and 3) high inherent sinterability. Green density was 62-63% of theoretical (2.62 gm/cc) density. Ball milling time required was 1.5 hours to reduce the apparent density from 17 gm/in^3 (as-received) to 13.5 gm/in^3 (desired density for pellet pressing). This powder sintered to 78% of theoretical density without the use of calcium carbonate ($CaCO_3$) as a sintering agent. One thousand nine hundred (1900) pounds of the powder were processed without $CaCO_3$ at an apparent density of 12 gm/in^3. Eight hundred (800) pounds were processed with 3/16 w/o $CaCO_3$ at an apparent density of 13.5 gm/in^3.

The base case $LiAlO_2$ powder processed in March was below average in sensitivity to $CaCO_3$. The powder was ball milled to an apparent density of 13.5 gm/in^3. This apparent density was sufficient to provide a sintered density of 74% of theoretical density. 1 to 1 1/4 w/o $CaCO_3$ was required to increase sintered density to 78% of theoretical. Normally a 4% increase can be achieved with 1/4 - 3/8 w/o $CaCO_3$.

Target Reactivity - J.E. Hansen

The average Li^6 content for all targets in the reactor, of a given $LiAlO_2$ case, must be within specified limits (± 2%). The specified limits and

Target Reactivity - J.E. Hansen (Con't)

accumulated Li^6 content of material produced are shown in Table 1. As shown in Table 1, the base case target production is well within limits, however, spike material must be adjusted to meet specifications.

Table 1

Status of Target Li^6 Control

$LiAlO_2$ Case	Specified Pile Average Li^6 Content mg Li^6/cc of $LiAlO_2$	Actual Average Li^6 Content of Shipped Production mg Li^6/cc of $LiAlO_2$
Base	6.07 ± 0.12	6.04
Spike	3.68 ± 0.11	3.92
Poison	13.84 ± 0.28 (12.26 ± 0.24 on revised specification)	None Shipped To Date

Sintering Time and Temperature - JE Hansen

Sintering conditions were revised from 17 hours at 1325°C to 14 hours at 1335°C. This revision was dictated primarily by the amount of throughput required. Sintering temperature is of concern only as it affects lithium loss. Analysis of material produced at the shorter time and higher temperature shows no significant change in lithium loss (this loss has averaged 2.5% over the program).

Aluminum Subassembly Oxidation - W.W. Olson

Aluminum subassemblies have in the past been furnace oxidized to minimize the possibility of zircaloy-aluminum bonding during reactor service. Evaluation of zircaloy and aluminum clads in autoclaved target assemblies has shown that this step can be eliminated in the process. The oxide coating developed on the aluminum and zircaloy internal surfaces during normal autoclaving is believed superior to that developed by the furnace oxidation step.

Ceramic Pellet Recovery - W.W. Olson

Recovery of the $LiAlO_2$ pellets from reject aluminum subassemblies has been performed in the past by mechanical removal of the aluminum cladding. Furnace melting of the aluminum from the pellets has been developed to a point where it can be used on a production basis. This procedure is considerably more efficient and pellet recovery is essentially 100% v.s. approximately 80% when mechanical methods are used.

Rigid Support Height Adjustment - W.W. Olson

Rigid support heights are established by grinding to develop the required annulus between the target and driver. Variations in etching times and

Rigid Support Height Adjustment - W.W. Olson (Con't)

the sizing and grinding operation generate some targets with rigid support heights below that required for standard driver fuel. A process has been developed for adjusting the rigid support heights to recover these targets. A die is used to upset the supports to a height within or above specifications. This procedure is simply cold forming of the support while attached to the target. Metallographic examination of support weld projections show no detrimental effect due to this upsetting operation. The only visual indication that upsetting has been performed is an indentation in the side of the rigid support. The support profile as seen by the coolant flow is unchanged.

MATERIALS DEVELOPMENT

Fluorine Contamination - $LiAlO_2$ - R.R. Studer

A number of $LiAlO_2$ powder lots have contained excessive amounts of fluorine in the as-received condition. This material was introduced into the process on the basis that a review of fundamental fluoride chemistry indicated the fluorine content of pellets should be reduced considerably during sintering. Subsequent analyses have shown that approximately 90% reduction of fluorine content is obtained during sintering. Therefore, no particular problem exists or is expected with fluorine in the sintered product.

Flame Sprayed Ceramic Product - R.R. Studer

Work on flame spray deposition of $LiAlO_2$ was initiated during the month. This work is directed at development of a process where the isotope level in the finished product can be controlled by the $LiAlO_2$ thickness. This thickness would be controlled simply by substituting various diameter rods of alumina or similar material. Deposition of as-received powder on a mild steel substrate was moderately successful. The deposit had fair adhesion to the steel with an estimated density of 60%. The main problem encountered was increasing roughness of the deposit as thickness increased, which was caused by nonuniform particle size. As-received and ball milled powders were deposited on an alumina tube. The as-received powder produced an open, rough deposit with poor adhesion. The ball milled powder gave better results, but adhesion still was not judged adequate. Microscopic examination of the material produced, using this technique, is now being performed and analytical samples have been submitted to determine lithium loss, if any, during flame spraying.

An attempt to spray a mixture of lithium carbonate and alumina powders to react to $LiAlO_2$ in the flame, was not successful. The powder was not free flowing and clogged the metering valves in the spray gun. If a coarser, more grannular carbonate can be obtained, this method of deposition will be further evaluated.

The problem of securing better adhesion to alumina calls for activating the surface of the alumina with a fluxing agent. There are numerous fluxes compatible with the end use of the targets, including several lithium salts.

[redacted]

Target Cladding Integrity - R.R. Studer

A different type of cladding defect was discovered by helium leak checking during the month. The outer surface appearance of the water path entry differed from that of earlier leakers. The defect appeared to be an inverted fold which had been observed earlier as originating only on the outer surface of clads made from K-reactor process tube stock. The attached photographs (Figures 1 through 5) may be compared with those in RL-GEN-1449. This type of defect is not likely to be discovered either by visual inspection as now practiced or by a fluorescent or die penetrant. Therefore, elimination of this kind of defect from the production stream is totally reliant on helium leak testing.

PROCESS DEVELOPMENT

Target Reactivity Control - J.E. Hansen

$LiAlO_2$ in the powder form is being tested in the 305 Reactor to determine the $Li^6/LiAlO_2$ ratio. Test capsules of powder are compared to a standard powder capsule to determine reactivity relative to the standard. The relative reactivity obtained is then plotted on a curve of reactivity values v.s. Li^6 content to determine the $Li^6/LiAlO_2$ weight ratio for the test powder. This test is judged capable of measuring the actual $Li^6/LiAlO_2$ weight ratio within ± 6%. The accuracy of $Li^6/LiAlO_2$ weight ratio determination by chemical and mass spectrographic means is ± 10%. Powder reactivity testing has been established at the following process locations: 1) prior to input into the process; 2) after mixing, if Li^6 content adjustments are required; and 3) after sintering. The after-sintering results are used to assign Li^6 values to finished target production.

Li^6 content in the powder is adjusted prior to fabrication as established by reactivity testing results. The Li^6 adjustment performed on 12 typical powder lots is shown in Table 2. The Li^6 content of the mixed powder attests to the mutual accuracy of the mixing operation and the 305 Reactor reactivity test results.

Table 2.

Powder Lot	Base Case As-Received Li^6 Content (% From Spec. Nominal)	Desired Li^6 Content (% From Spec. Nominal	Deviation (% From Specification Nominal)
A-333	+ 6.7	0	+1
A-134	+13.1	0	-2.6
A-135	+10.0	0	-0.3
A-136	+11.1	0	-0.9
A-137	+16.2	0	-0.0
A-138	+10.0	0	+0.4
A-139	+10.8	0	+0.1
A-141	+19.9	+5	+0.6
A-142	+13.4	+5	+1.2
A-143	+13.5	+5	-0.1
A-144	+ 9.4	+5	+1.4
A-145	+11.1	+5	-1.1

$\bar{X}$ = +0.05%

Target Reactivity Control - J.E. Hansen (Con't)

The Li^6 content of $LiAlO_2$ pellet production is assigned following sintering. This assignment cannot be made at an earlier point in the process since changes in the $Li/LiAlO_2$ weight ratio, and therefore the $Li^6/LiAlO_2$ weight ratio, occur during sintering.

Control of Li^6 content on a per column basis is determined at the driver-target assembly station. Targets containing excess Li^6 above the specified upper column limit are identified with a stamped plus sign (+). Those containing Li^6 within the column limits are not identified in any particular manner. Those targets containing less Li^6 than the specified lower column limit are identified with a stamped minus sign (-). Targets without supplemental identification can be charged in any column. Those with + or - signs must be charged in pairs to maintain the column average within specified limits.

Control of target element Li^6 content on a overall reactor basis requires periodic adjustment of input powder. The Quality Control Operation maintains an accumulating average Li^6 content for all target elements shipped. Li^6 adjustments are made in the input powder as this average deviates from specified values. This relationship closes the Li^6 control loop.

The control procedures to maintain target reactivity at specified levels is graphically illustrated in Figure 6.

C H Shaw

Manager,
Special Products Engineering Operation
N-Reactor Fuels Section
Hanford Atomic Products Department

CH Shaw:jan

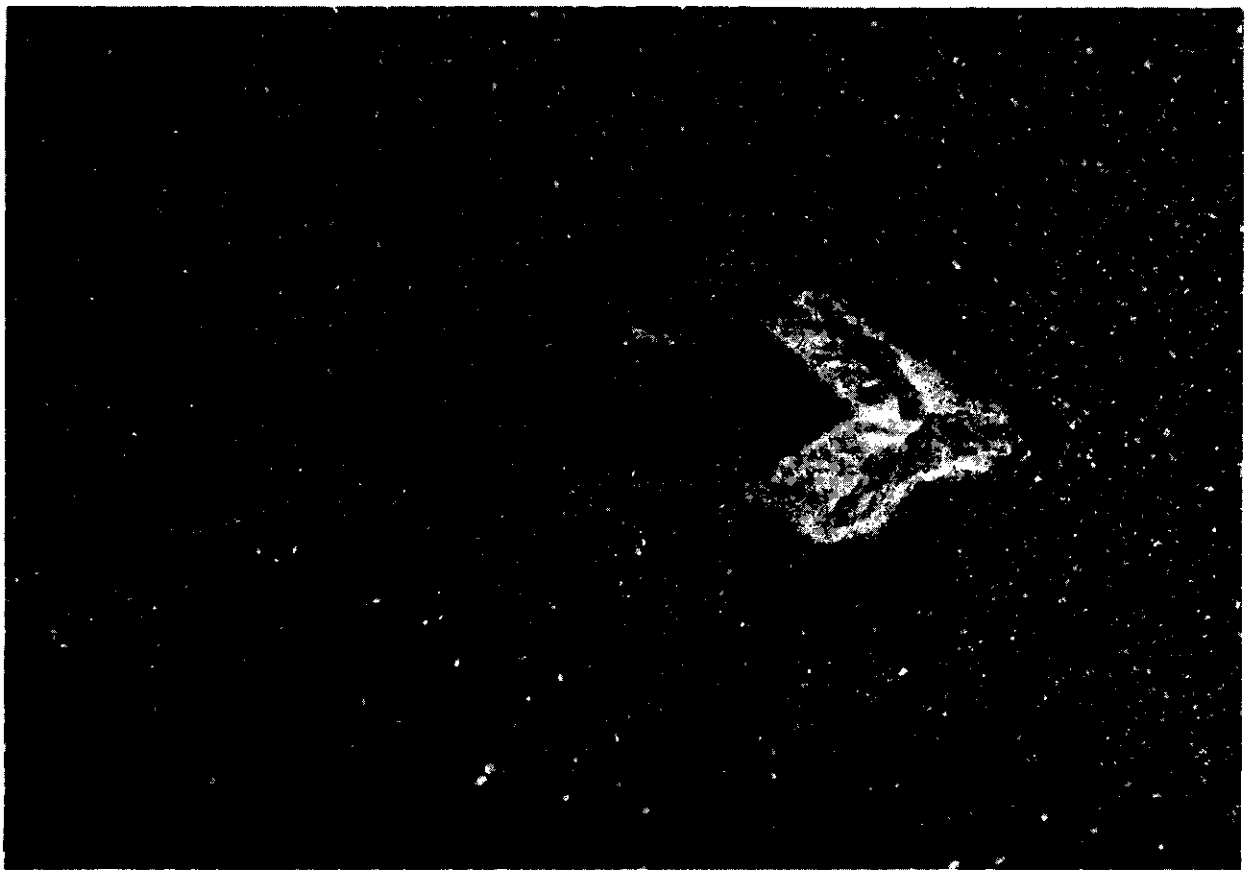
Attachments (Figures 1-6)

567 5470 A 20X

FIGURE 1

Outer Surface Indication of Water Path

(Target S-02757)

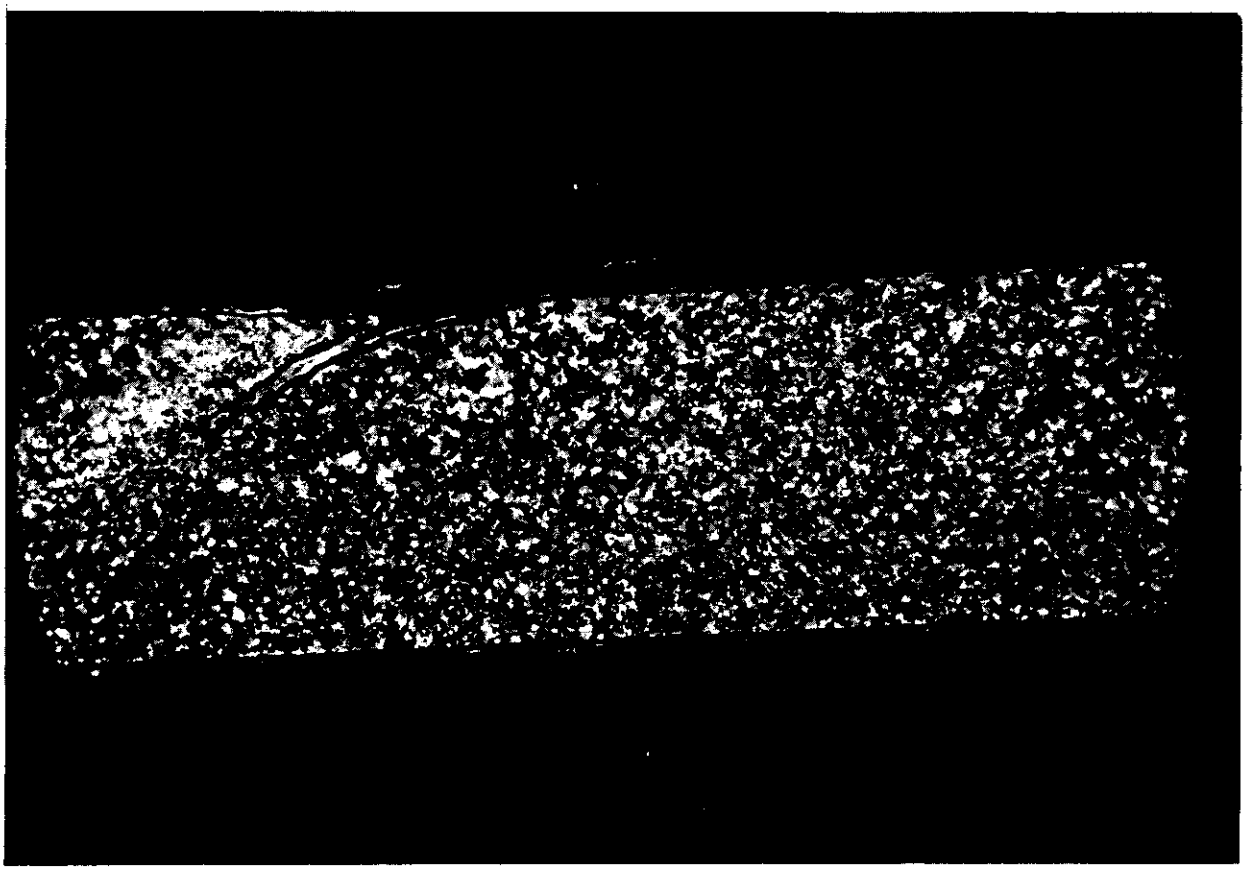


567 5470 B 20X

FIGURE 2

Fold on inner surface of clad, analagous to O.D. defects causing earlier leakers. Note secondary portion of defect immediately behind longer barb of "arrowhead".

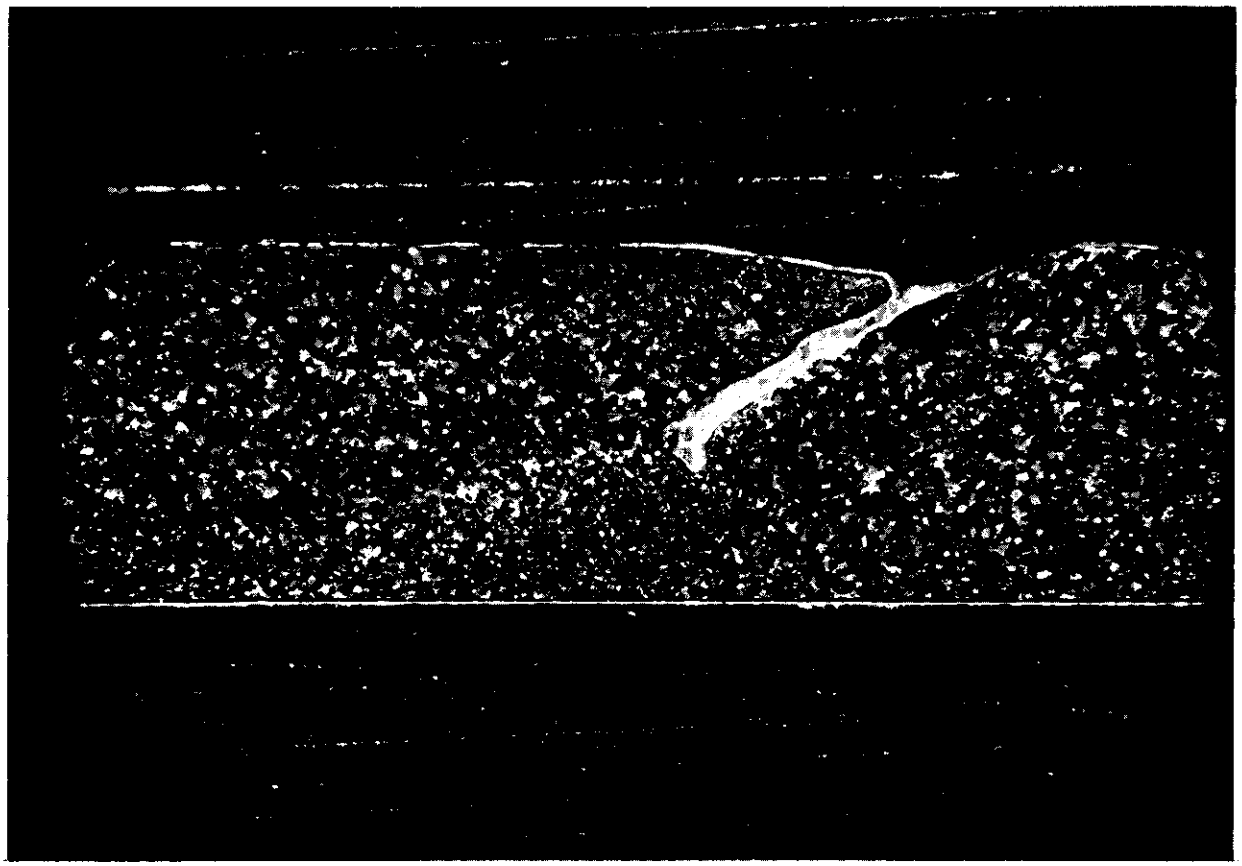
(Target S-02757)



567 5470 C 50X

FIGURE 3

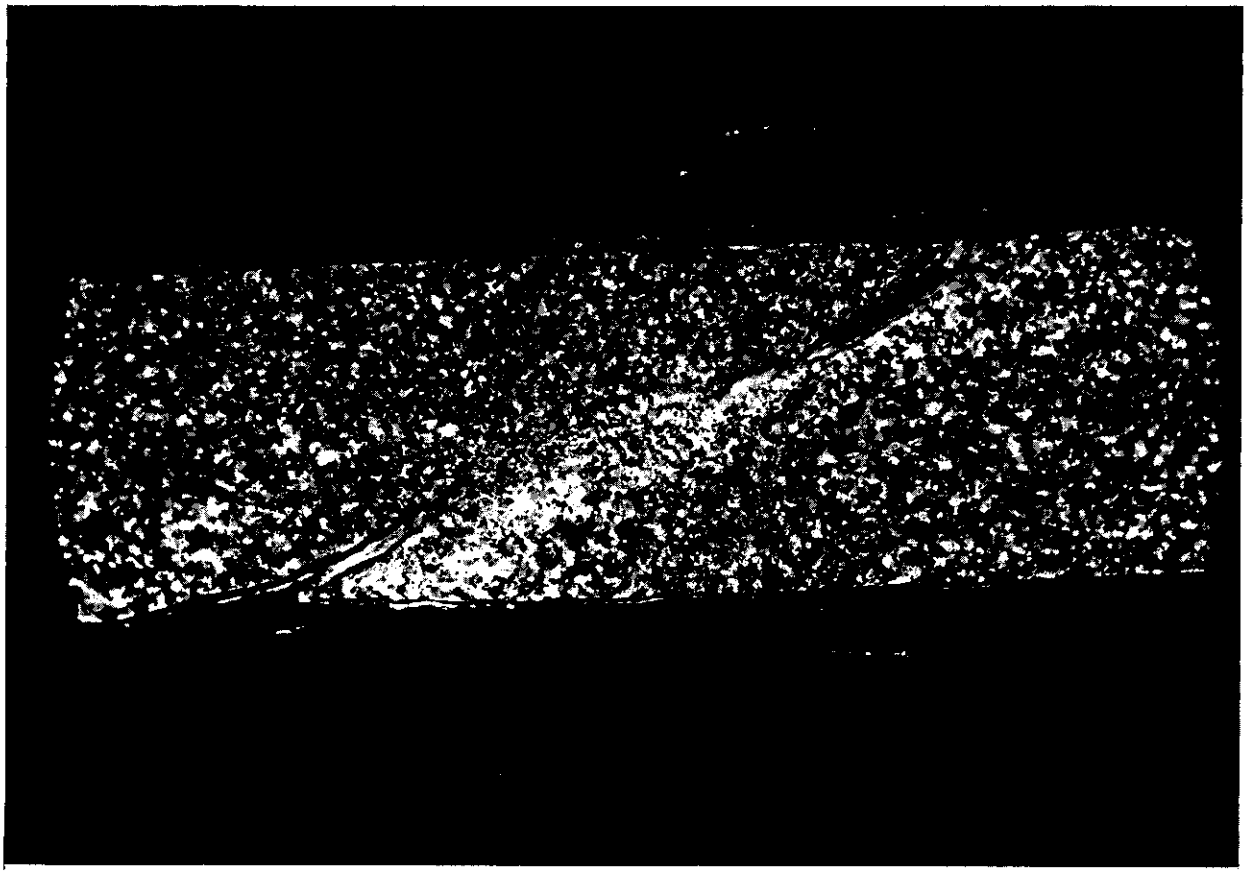
Section through defect zone, approximately in axial alignment with small secondary defect showing in Figure Number 2.



567 5470 C 50X

FIGURE 4

Section Midway Between End of "Barb" and "Point"



567 5470 D 50X

FIGURE 5

Section after polishing approximately 15 mils from surface of Figure 4. Water path has been polished through, but disturbed metal adjacent to fold is visible.

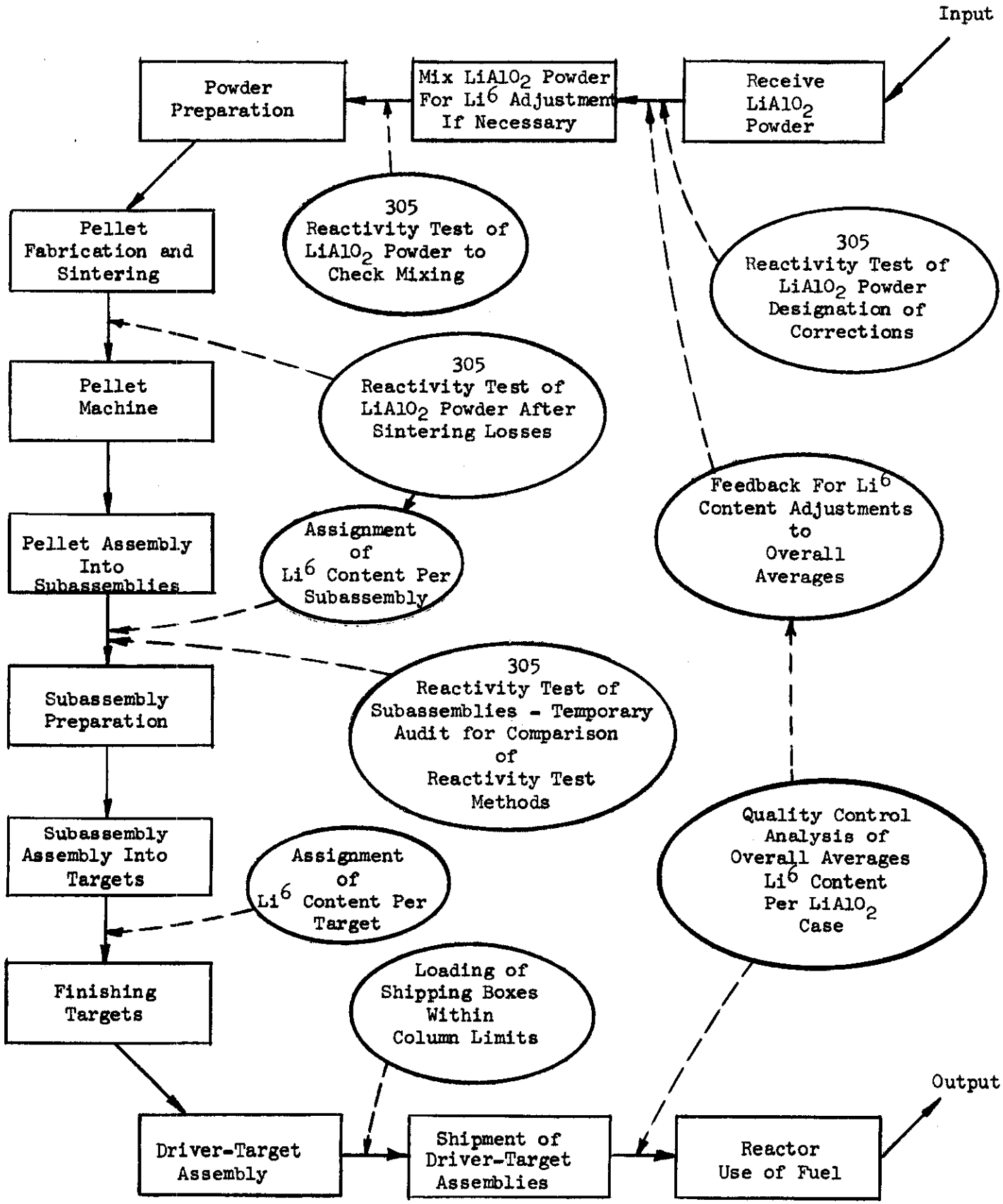


Figure 6

Target Process Reactivity Control Chart