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MARK 60B BLANKET TUBE FAILURE (U)

by

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August 1, 1975

TO: J. P. MALONEY
 RAW MATERIALS TECHNOLOGY SECTION

Classified By R. R. Herries
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 CG 303.8

FROM: M. K. CARLSON *MKL*
 RAW MATERIALS TECHNOLOGY SECTION

MARK 60B BLANKET TUBE FAILURE

Following documents a talk given at AED-Wilmington on July 18,
 Copies of slides used in that talk are attached.

INTRODUCTION

A Mark 60B lithium-aluminum target element jammed during discharge from 105-K reactor on March 17, 1975. This report includes a description of the failure and how fragments from it were analyzed to determine the most probable mechanism by which failure occurred. This failure mechanism was duplicated in special tests to confirm the conclusions and help develop a preventative program.

SUMMARY

The failed target element was single extruded. When a piece of lithium rich dross fell into the mold during casting, it remained in tact until coextrusion when it penetrated or greatly thinned the cladding. The inclusion reacted with moderator during irradiation to corrode the target element and cause swelling, which ultimately caused jamming.

DISCUSSION

The Mark 60B blanket tube, NK 1363C-2, jammed during discharge after being raised about 7 feet. It was lowered about 2 feet when it stuck again. The tube was finally removed with the stainless steel semi-permanent sleeve. Cladding was stripped off about 4 feet of the tube exposing corroded Li-Al core. Large shreds of cladding protruded from under the semi-permanent sleeve which obscured the top 4 or 5 feet of the tube. The bottom half of the tube appeared normal, as did 15 other Mark 60B's including one with a core from the same casting as the failed tube.

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August 1, 1975

Since nothing unusual prior to discharge was noted in 105-K, this investigation centered almost entirely on the 300 Area processing.

Over 90% of the Li-Al targets produced are double extruded, such as the Mark 16 inner target. The pre-extrusion step helps break up and string out inclusions so that they are less likely to cause cladding penetrations during coextrusion. Mark 60B targets were not pre-extruded because the large coextrusion core diameter required (6.180") would have allowed a pre-extrusion ratio of only about 1.4 to 1 using existing 8" diameter containers. That ratio is too low to be effective in breaking up inclusions.

Lithium-Aluminum alloys are normally made by mechanically dropping ~3 pound aluminum ingots and ~1/2 pound cans of lithium into a graphite crucible inside a vacuum chamber. The heat from which the failed tube came (NK 1363) was charged by hand with scrap Li-Al from rejected Mark 60B cores and castings. These scrap pieces probably weighed from 7 to 15 pounds and could have chipped dross from the sides of the crucible as they were charged. The crucible had been used for 27 heats so it was surely covered with a thick layer of dross, which is a solid reaction product of the melt with the atmosphere.

Two unusual quality control test results were noted when reviewing records of the failed tube. An unusual bond test trace was produced but was marked ok. It should have been retested and rejected if the trace was reproduced. A similar trace was produced in a recent test by combining a large nonbond standard (3' x 1/2") with mechanical mismatch between recorder and target rotation. Nuclear test gage results indicated an area of high lithium content, giving the failed tube the highest NTG spread of all tubes produced in that run. There was no specification for allowable spread and the 7.4% spread for NK 1363C-2 was not thought to be extraordinary at the time.

Fragments from the failed tube were of two types; small pieces of heavily corroded Li-Al core and pieces of outer cladding up to 3" wide by 12" long with core material bonded to the inner surface.

Analyses of fragments showed that the cladding was 1100 aluminum alloy as specified and it has normal mechanical properties. Li-Al core material was bonded to cladding and had the expected chemical and isotopic composition. It was hard and brittle as expected, because of the presence of tritium and helium.

Core fragments were about 80% oxide and the oxide was a type (α - $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) that forms after a long time (>1000 hours) in pH5 water or in a caustic solution (pH >9).

The inner surface of the outer cladding was chemically attacked in small areas where there was no core material bonded to it, indicating the presence of a corrosive substance inside the tube. Analyses of fragments from the failed tube support the conclusion that a lithium rich dross inclusion was the most probable cause of failure.

August 1, 1975

The conclusion was tested by putting dross inclusions in six Mark 16 inner target cores and processing them through NTG. A variety of surface appearances resulted at various stages of the operation, ranging from normal appearance to pitted. The bond tester picked up only the worst defect while the NTG detected all 6.

The fluoroscope and eddy current tester have not been used to detect inclusions in targets in the past. Both devices detected large inclusions in the six special tubes. Subsequent work showed the fluoroscope to be much more sensitive than the eddy current tester.

Corrosion tests on the six special tubes showed that the dross was reactive and caused enough swelling to rupture cladding. Even if pin holes were over dross that was not very reactive, the simulated moderator worked its way to more reactive dross and cladding eventually ruptured.

NTG and bond test records of tubes produced this year were reviewed. None was similar to records for the failed target. The tube with the highest NTG spread (5.7%) was sectioned and no defect was found.

Five single extruded tubes from the Building 320-M honeycomb and 3 tubes (from heat NK 1363) recalled from reactor storage were fluoroscoped. Two of the tubes (one from heat NK 1363) had cladding penetrations caused by dross-graphite inclusions. An additional tube with a cladding penetration was found during a routine metallurgical laboratory examination. All of these tubes had acceptable NTG and bond test results. Because the NTG and bond test are not as sensitive as the fluoroscope, there is no positive way to identify potential failures in the reactor.

CONCLUSIONS

1. A dross inclusion was the most likely cause of failure.
2. Dross and graphite inclusions can be detected by existing equipment.
3. Casting procedures should be improved.
4. All targets should be double extruded.

RECOMMENDATIONS

1. The failed Mark 60B target should not be examined. The most probable cause of failure has been determined and further examination would require separating the semi-permanent sleeve from the tube to determine if a foreign object (such as a nut or bolt) caused jamming. Even if that occurred, there is no evidence to support extensive corrosion of Li-Al in moderator unless a reactive inclusion had been present.

The failed target is in a special container in the disassembly basin. Water will be pumped from the container into a drum for shipment to a waste tank. The container and target will then be processed by Separation Department.

2. Continue irradiation of targets in reactors, since there is no evidence (based on NTG or bond tests) of any defects in them.

PROGRAM

Short range programs involve use of available inspection equipment to detect inclusions in targets and improving casting techniques. Temporary operating procedures for fluoroscopy and eddy current testing targets have been written and used. New 300 Area NTG acceptance criteria are being developed by RMD Quality Control.

Production records of targets in reactors are being reviewed to verify that all were acceptable. Records of single extruded targets produced this year have been reviewed. These targets comprise the majority of single extruded elements in reactors. Some extra targets from previous runs were stored in 100-Areas and charged recently, so their records also need to be reviewed.

Lithium-Aluminum vacuum furnace casting procedures and techniques are being reviewed and have been improved. Crucible usage is being limited to 20 heats, pouring rates have been slowed and "capping" (pouring extra metal on a casting that has started to solidify) has been stopped. Scrap charges will be limited to small pieces of extruded Li-Al that can be charged mechanically with aluminum ingots.

Long range programs are to double extrude all target elements and air melt lithium-aluminum alloys.

Double extrusion requires no new technology since it is the predominant process in Building 320-M now. Thirteen inch diameter castings will be needed to achieve pre-extrusion ratios capable of breaking up inclusions in material that is now single extruded. A considerable amount of new equipment will be needed to handle castings of that size in Building 320-M.

Pre-extrusion breaks up inclusions so that they are less likely to cause cladding penetrations during coextrusion. To solve the inclusion problem, the casting process must be modified. Air melting lithium-aluminum will allow greater access to melts than is possible with the present vacuum melting system. Dross and graphite can be skimmed from the melt before pouring and the crucible can be scraped clean after every heat. Lithium-Aluminum has been air melted successfully in tests, but some techniques need to be worked out and lithium loss factors need to be established before it can be used as a production process.

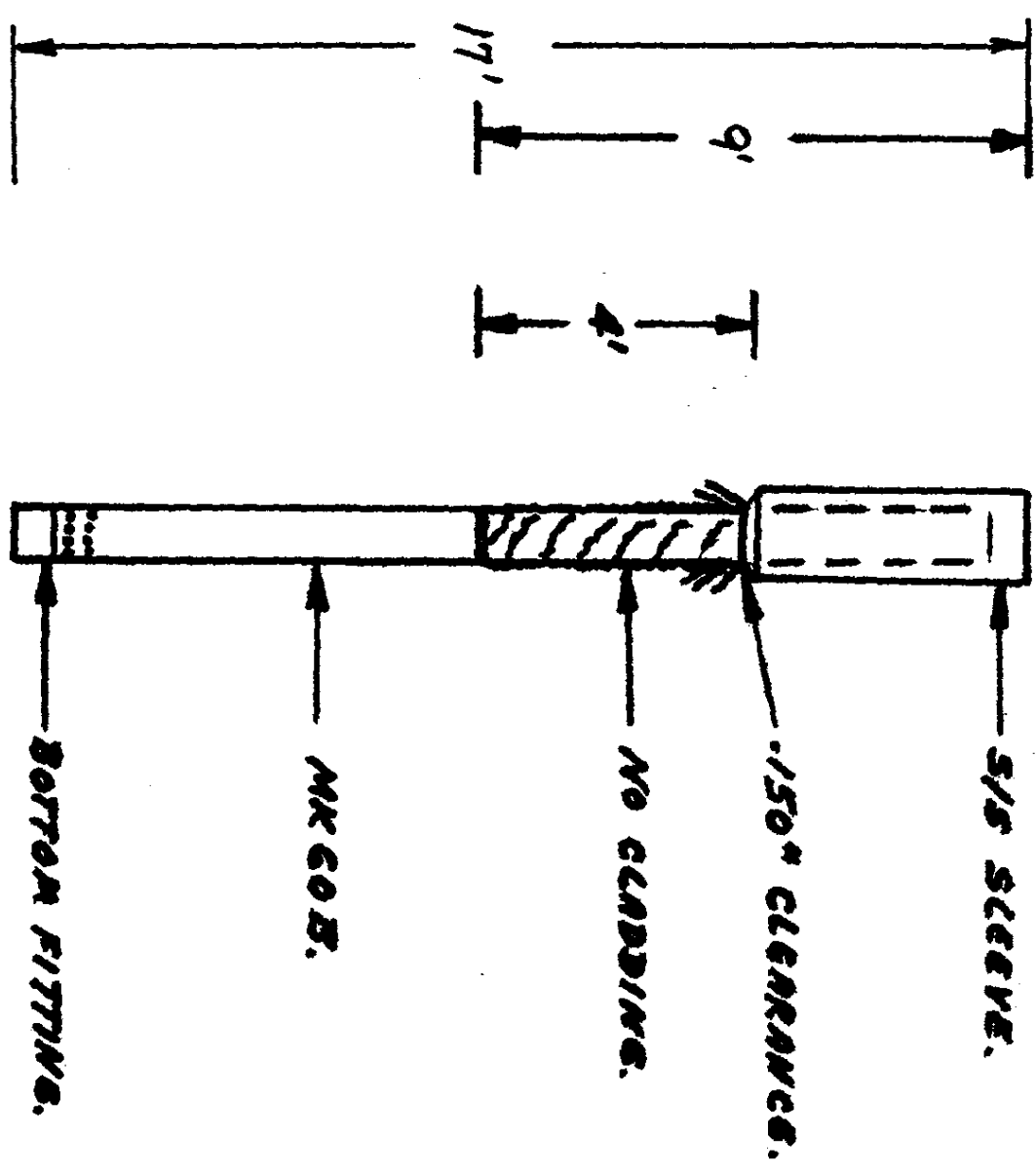
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MARK 60B BLANKET TUBE FAILURE

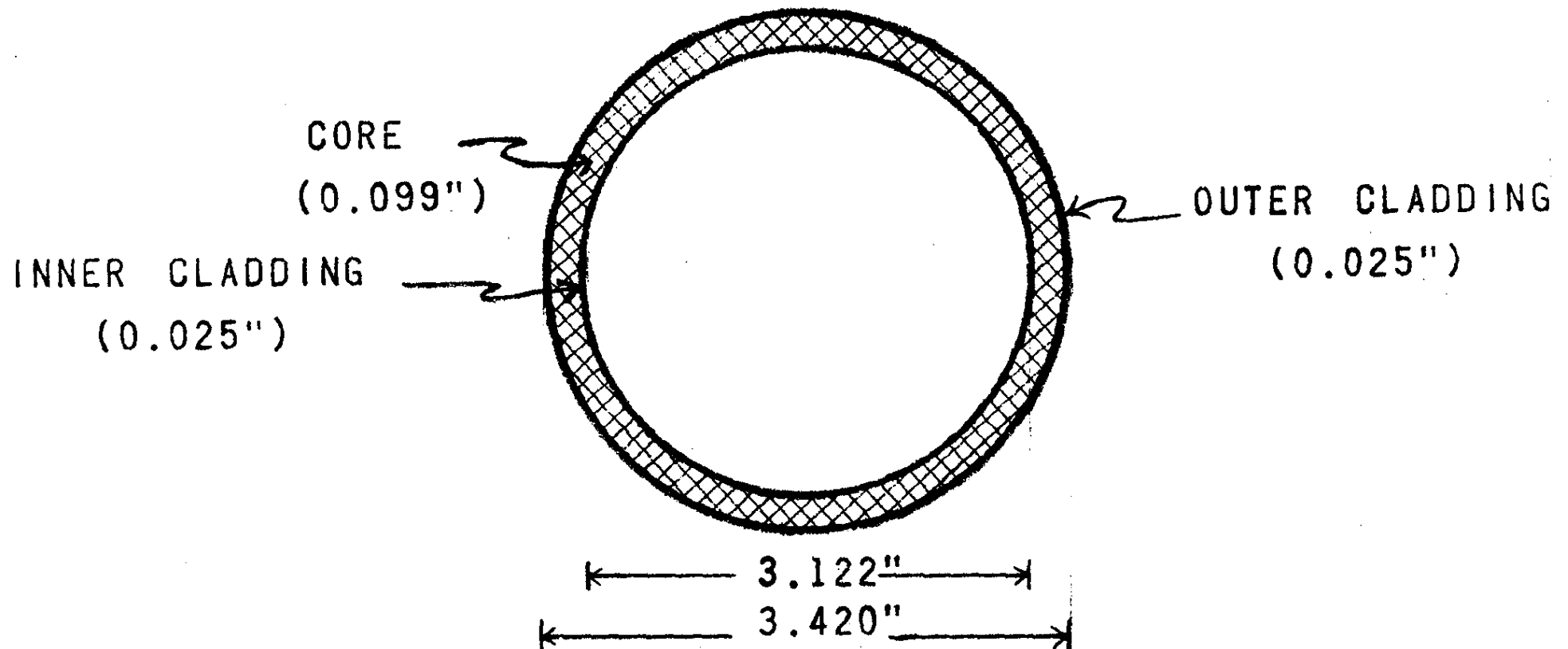
- o FAILURE DESCRIPTION
- o ANALYSIS OF FRAGMENTS
- o PROBABLE MECHANISM
- o DUPLICATION OF FAILURE
- o CONCLUSIONS
- o PROGRAM
- o RECOMMENDATIONS

SUMMARY

- o TUBE WAS SINGLE EXTRUDED
- o LI RICH DROSS INCLUSION PENETRATED CLADDING AND CAUSED FAILURE
- o SIMILAR INCLUSIONS WERE FOUND IN OTHER SINGLE EXTRUDED MATERIAL ON HAND
- o FAILURE WAS DUPLICATED IN CONTROLLED TESTS
- o FLUOROSCOPE WILL DETECT DROSS INCLUSIONS
- o PROCESS CHANGES WILL IMPROVE TARGET QUALITY
 - ✓ CASTING TECHNIQUE
 - ✓ DOUBLE EXTRUSION



MARK 60B TARGET



100 AREA BACKGROUND

BLANKET TUBE - NK 1363C-2

220 DAYS IN REACTOR

NOTHING UNUSUAL

MODERATOR ANALYSES - OK

GAS VOLUME RATIO = 40

40% BURNUP

15 OTHER MARK 60B's (INCLUDING NK 1363 C-1)
APPEARED NORMAL

TARGET PRODUCTION

- 10 -

DPSP-75-1219

MK 16 IT

MK 60B

CAST



3.41:1 EXTRUDE
TO LOG

X 10 MACHINE
TO CORE

X 2

35.1
(120:1) COEXTRUDE
TO TUBE

17:1
(17:1)

COEXTRUDE

TO TUBE

300 AREA BACKGROUND

- o FIRST TIME 60B PRODUCED
 - ✓ 2.1% LI
 - ✓ 44.3% 6LI
- o CAST CORE VS DOUBLE EXTRUDED
- o CLEAN MK 60B SCRAP CHARGE
- o GRAPHITE CRUCIBLE - 28TH HEAT
- o NORMAL IMPURITY LEVELS
- o NO PRODUCTION PROBLEMS

300 AREA BACKGROUND (CONTINUED)

o BOND TEST - ULTRASONIC GAP TEST

✓ UNUSUAL TRACE WAS MARKED OK

✓ DUPLICATED BY DEFECT 3' LONG x 1/2" WIDE

o NUCLEAR TEST GAGE (NTG) RESULTS NK 1363C-2

4.13, 4.44, 4.21, 4.14, 4.15 G⁶LI/FT

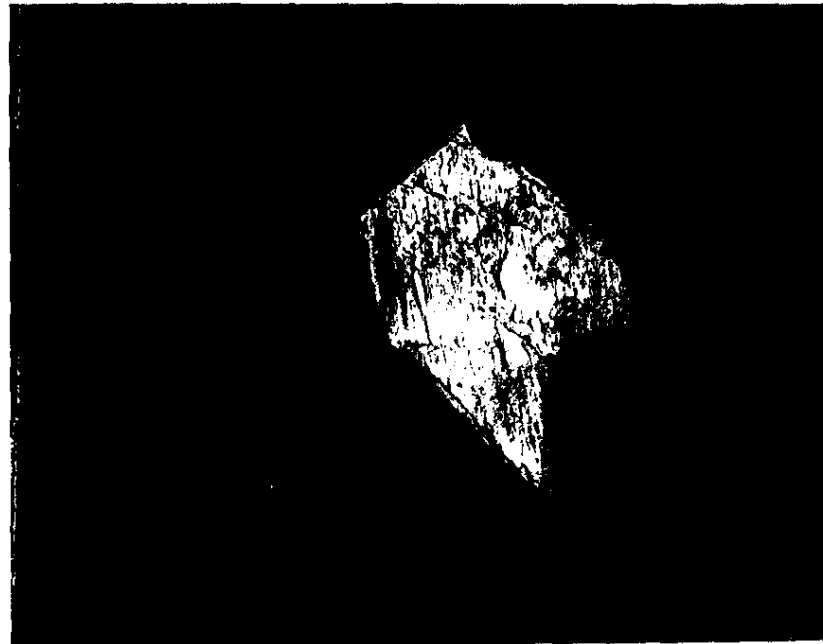
✓ NK 1363C-2 7.4% SPREAD

✓ NEXT HIGHEST 3% SPREAD

✓ MOST TUBES 1% SPREAD



Outer Cladding
Outer Surface
Negative No. EE-55154M



Outer Cladding
Inner Surface
Negative No. 55155M

Outer Cladding With Core Bonded To Inner Surface
Approximately 1/2 x



Approximately 5 x

Negative No. EE-55126M

Corroded Li-Al Flake From Core Of Failed Mark 60B
Blanket Tube.

NORMAL ANALYTICAL RESULTS

			TEST
o CLADDING	✓	1100 ALUMINUM	(CHEM)
	✓	NORMAL MECHANICAL PROPERTIES	(HARDNESS)
	✓	NORMAL EXTERNAL CORROSION	(X-RAY)
o CORE	✓	NO SIGNIFICANT UNUSUAL ELEMENTS	(CHEM)
	✓	1.1% LI AFTER IRRADIATION	(CHEM)
	✓	$^6\text{Li} : ^7\text{Li}$ 1:3	(CHEM)
	✓	CORE BONDED TO CLADDING	(MET)
	✓	CORE HARD AND BRITTLE	(HARDNESS)

CORE MATERIAL



Approximately 100 x

Negative No. EE-55188M

Chemical Attack On Outer Cladding Inner Surface.

UNUSUAL ANALYTICAL RESULTS

o CORE

✓ SAMPLES 80% OXIDE

✓ OXIDE OF TYPE FORMED AFTER LONG
TIME IN PH 5 WATER OR IN SOLUTION \geq PH 9

✓ OXIDE VOLUME 3X ALUMINUM VOLUME

o CLADDING

✓ INNER SURFACE ATTACKED

TEST

(MET)

(X-RAY)

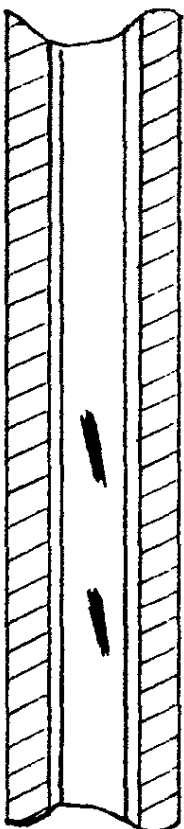
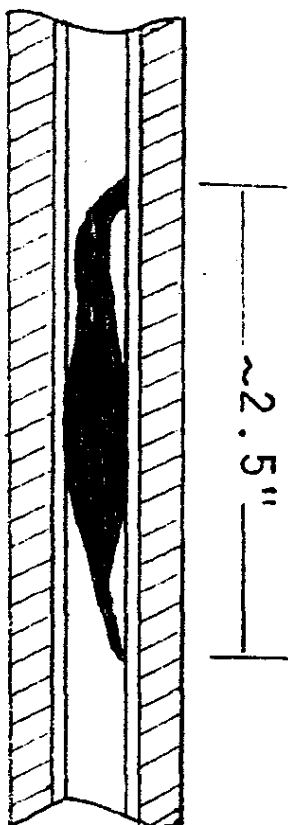
(MET)

INITIAL CONCLUSIONS

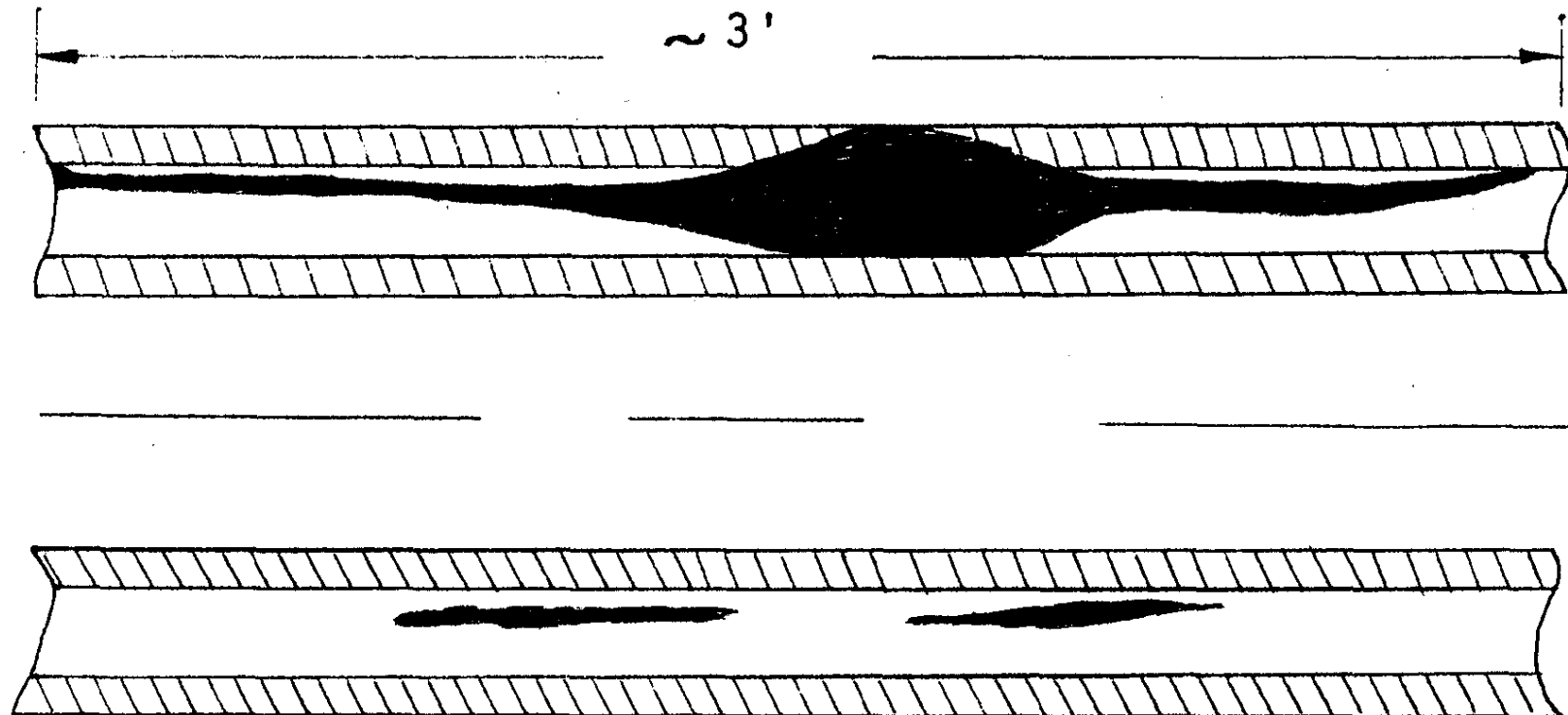
- | | |
|---|-------------|
| 1. ULTRASOUND DISRUPTING DEFECT PRESENT | TEST |
| 2. HIGH NEUTRON ABSORBER PRESENT
NO UNUSUAL ELEMENTS
∴ MORE LITHIUM PRESENT THAN NORMAL | BOND TEST |
| 3. CORE EXPOSED TO MODERATOR LONG TIME
AND/OR EXPOSED TO CAUSTIC | NTG
CHEM |
| 4. CLADDING INNER SURFACE CORRODED | X-RAY |
| 5. SWELLING OCCURRED AND CAUSED JAMMING | MET |
| | ASSUMED |

PROBABLE MECHANISM

1 5% - 10% LI DROSS POURED WITH CASTING



2 CLADDING PENETRATED OR GREATLY THINNED BY DROSS
OR GRAPHITE INCLUSION



- 3 DROSS, CONTACTED BY MODERATOR, DISSOLVED AND FORMED CAUSTIC SOLUTION, WHICH ATTACKED CORE AND CLADDING
- 4 SWELLING CAUSED BY CORROSION PRODUCT AND GAS EVOLUTION

DROSS TUBE APPEARANCE

TUBE	DROSS, GRAMS	EXTRUDED	CLEANED	DRAWN & STRAIGHTENED
1	13	RIPPLES	3 PITS	OK
2	11	RIPPLES	MANY PITS	SMEARED PITS
3	18	RIPPLES & HOLES	MANY PITS	SMEARED PITS
4	14	OK	OK	OK
5	9	OK	MANY PITS	SMEARED PITS
6	5	OK	SOME PITS	SMEARED PITS

NONDESTRUCTIVE TEST RESULTS

TUBE	BOND TEST, IN.	NTG SPREAD, %	FLUOROSCOPE (INCHES)	EDDY CURRENT
1	0	7.0	1/2 x 22	28
2	0	3.9	1 x 24	36
3	1/2 x 7	13.7	1/2 x 11	100
4	0	2.3	1 x 17	40
5	0	3.8	1 x 14	90
6	0	3.1	1/3 x 19	10



Approximately 2 x

Negative No. EE-55477M

Tube Number 3 Before Corrosion Testing



Approximately 1/2 x

Negative No. EE-55479M

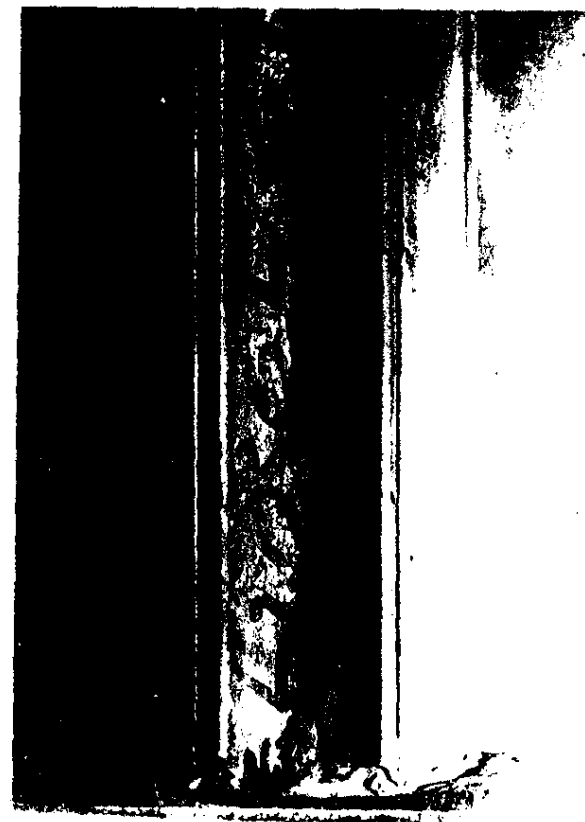
Tube Number 3 After 18 Hours Exposure To 95°C pH 5 Water.



13 Days
Negative No. EE-55522M



16 Days
Negative No. 55538M



24 Days
Negative No. 55556

Tube Number 1 After Being Exposed To 95° pH 5 Water
For Various Times. Approximately 1/2 x.

SINGLE EXTRUDED TUBE STATUS
RE-INSPECTION OF SINGLE EXTRUDED TUBES

o INSPECTED 8 TUBES

✓ 3 CLADDING PENETRATIONS (DROSS & GRAPHITE)

✓ 1 HIGH NTG SPREAD (5.7%) - NO DEFECT

o TUBES IN REACTOR

ELEMENT	NO.	NTG	BOND TEST
MK 18A (PK)	65	NONE	OK
MK 41 IT (HT)	30	OK	FAIR
MK 60B (NK)	97	OK	OK

o THEREFORE, NO POSITIVE WAY TO IDENTIFY ANY
POTENTIAL FAILURES IN REACTOR

CONCLUSIONS

- o DROSS INCLUSION MOST LIKELY CAUSE OF FAILURE
- o DROSS AND GRAPHITE INCLUSIONS CAN BE DETECTED WITH EXISTING EQUIPMENT
- o CASTING PROCEDURES SHOULD BE IMPROVED
- o ALL TARGETS SHOULD BE DOUBLE EXTRUDED

RECOMMENDATIONS

- o NO FURTHER EXAMINATION OF FAILED 60B
- o CONTINUE IRRADIATION OF TUBES IN REACTOR

PROGRAM

o SHORT RANGE

- ✓ FLUOROSCOPE ALL SINGLE EXTRUDED TARGETS
(321-TOP-168)
- ✓ EDDY CURRENT TEST ALL TARGETS (321-TOP-169)
- ✓ TIGHTEN NTG SPREAD
- ✓ REVIEW PRODUCTION RECORDS
- ✓ EVALUATE PROCEDURES
- LIMIT CRUCIBLE USE
- CHARGING METHOD & SIZE

o LONG RANGE

- ✓ DOUBLE EXTRUDE ALL ELEMENTS
 - ✓ AIR MELT LI-AL
- 