

NAA-SR-2570

COPY

MASTER

BEHAVIOR OF STRUCTURAL MATERIALS
EXPOSED TO AN
ORGANIC MODERATED REACTOR ENVIRONMENT

AEC Research and Development Report



ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

BEHAVIOR OF STRUCTURAL MATERIALS
EXPOSED TO AN
ORGANIC MODERATED REACTOR ENVIRONMENT

BY:

N. J. GIOSEFFI
H. E. KLINE

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.
P.O. BOX 309 CANOGA PARK, CALIFORNIA

CONTRACT: AT(11-1)-GEN-8
ISSUED: OCT 1 1959



DISTRIBUTION

This report has been distributed according to the category "Metallurgy and Ceramics" as given in "Standard Distribution Lists for Unclassified Scientific and Technical Reports" TID-4500 (14th Ed.), October 1, 1958. A total of 605 copies was printed.

ACKNOWLEDGMENT

The authors wish to acknowledge the cooperation and helpful suggestions of the OMRE operating personnel, under the direction of Dr. C. A. Trilling. The contributions of K. H. Campbell and D. R. Muller are especially appreciated.

The efforts of A. B. Fenley and D. Jones of the Materials Development Group are acknowledged in the preparation, handling, and testing of specimens, as well as many additional activities related to the project.

The authors gratefully acknowledge the services of R. J. Burian and J. E. Gates of Battelle Memorial Institute who directed the hot cell work associated with the postirradiation examination of the in-pile specimens.



CONTENTS

	Page
Abstract	v
I. Introduction	1
II. Procedures	2
A. Specimen Material Selection	2
B. Specimen Preparation	2
C. Specimen Loading	4
D. Exposure Conditions	7
E. Postexposure Examination	11
III. Discussion of Results	23
IV. Conclusions	26
Appendixes	
A. Techniques of Neutron Dosimetry Analysis	A-1
B. Corrosion and Mechanical Properties of Materials Exposed in the OMRE	B-1
References	Ref-1

TABLES

I.	Corrosion Specimens in the OMRE	3
II.	In-Pile Weight Changes	16
III.	Out-of-Pile Weight Changes	21
IV.	OMRE Exposure - Summary of Room Temperature Tensile Properties	24
V.	OMRE Exposure - Summary of Room Temperature Impact Properties	25
B-I.	Weight Percent Composition of Materials Tested	B-3
B-II.	Corrosion Study Tensile Data	B-4
B-III.	Corrosion Study Impact Data	B-5
B-IV.	Corrosion Test Results on Materials Exposed in OMRE Bypass Line	B-6



FIGURES

	Page
1. Typical Specimens Used in OMRE Corrosion Study (7500-4710).	2
2. OMRE Dummy Element Specimen Holder (9561-5146A)	4
3. Inserting Specimens in an OMRE Dummy Element (9561-5146B)	5
4. In-Core Location of Corrosion Study Elements (9561-5452)	6
5. Bypass Specimen Holder Assembly (9561-5148) (7500-5105).	8
6. Location of Specimens in OMRE Bypass (9561-4704A) (9561-4704B)	9
7. Exposure Conditions in the OMRE (Power Level and Exposure).	10
8. Exposure Conditions in the OMRE (Bulk Coolant Temperature and HB Content).	10
9. Specimen Holder Shortly After Removal From OMRE Dummy Fuel Element (7500-4734)	12
10. Relative Gamma Ray Intensity Curve of Aluminum - 0.5 wt % Cobalt Dosimeter Wire (A-30383)	13
11. Relation of Integrated Flux as Determined From Dosimeter Analysis and the Relative Gamma Ray Intensity (A-30384)	13
12. Neutron Flux Distribution Determined by Radiochemical Techniques .	14
13. Control Specimens after Cleaning in Lestoil (RM11916) (RM11917). .	15
14. Postexposure Appearance of In-Pile Corrosion Specimens (HC1857) (HC1859) (HC1850) (HC1851)	17
15. Bypass Specimen Holder Assembly after Removal (7500-4724)	19
16. Corrosion Specimens Exposed in OMRE Bypass Line (7500-4726D) (7500-4726C).	20
17. U-Bend Specimens Exposed in the OMRE Bypass Line (7500-4726A) (7500-4726B).	21



ABSTRACT

The behavior of various structural materials, including Type 1020 carbon steel, Types 304 and 410 stainless steel, Type 4130 alloy steel, aluminum, and magnesium, in an organic moderated and cooled reactor have been determined.

In-pile specimens examined were exposed to measured integrated neutron fluxes in the order of 9.0×10^{19} nvt thermal and 2.8×10^{20} nvt fast (energies > 1 Mev). Exposure time was eight months, during which the reactor achieved 544 Mwd of operation. A polyphenyl, composed of ortho-, meta-, and para-terphenyls and diphenyl, with various amounts of high boiler compounds, served as the coolant. Nominal bulk coolant temperature in the core was 600°F. Out-of-pile specimens were exposed for 13 months in a bypass line of the reactor.

Results of the examination indicated the environment to be noncorrosive in nature. With the exception of magnesium, the corrosion resistance of all materials tested was considered good to excellent. The environment caused neither stress corrosion nor accelerated attack of bimetal couples. Irradiation had no significant effect on the corrosion resistance of the materials tested.

Mechanical property changes after irradiation were the same as those expected in materials irradiated without the polyphenyl environment. These changes were attributed to radiation damage. Ferritic grade steels were most affected; however, the magnitude of these changes is not considered serious. Slight changes in the postexposure mechanical properties of out-of-pile specimens were concluded to be thermally induced.



I. INTRODUCTION

The use of polyphenyls as moderator-coolants offers an attractive concept in nuclear power reactors. A major advantage is the high corrosion resistance of many common structural materials in the polyphenyls. Consequently, a wide choice of structural and fuel cladding materials is made possible. Data obtained in the NAA-20 MTR test loop¹ and other investigations^{2,3,4} were considered, prior to the selection of materials for this investigation.

The work described herein is part of a corrosion program being conducted by Atomic International, in support of research and development of the organic moderated and cooled power reactor concept. The purpose of the program is to obtain corrosion test data for structural and cladding materials in hot polyphenyls.

When investigating materials for construction of a nuclear reactor, the effects of radiation on mechanical and corrosion resistance properties must necessarily be considered. The Organic Moderated Reactor Experiment (OMRE)⁵, being operated for the AEC by Atomic International at the National Reactor Testing Station in Idaho, made possible the investigation of material compatibility under realistic reactor operating conditions. In-pile exposures were made in two dummy fuel element locations, and out-of-pile exposures were made in an existing section of a bypass line.



II. PROCEDURES

A. SPECIMEN MATERIAL SELECTION

Materials exposed included Type 1020 carbon steel, Types 304 and 410 stainless steel, AISI 4130 alloy steel, aluminum, and magnesium. The in-pile specimens were essentially duplicated in the out-of-pile, bypass loading. A detailed listing of materials and number of in-pile and out-of-pile specimens is given in Table I.

B. SPECIMEN PREPARATION

Typical specimens used in the OMRE corrosion study are shown in Figure 1.

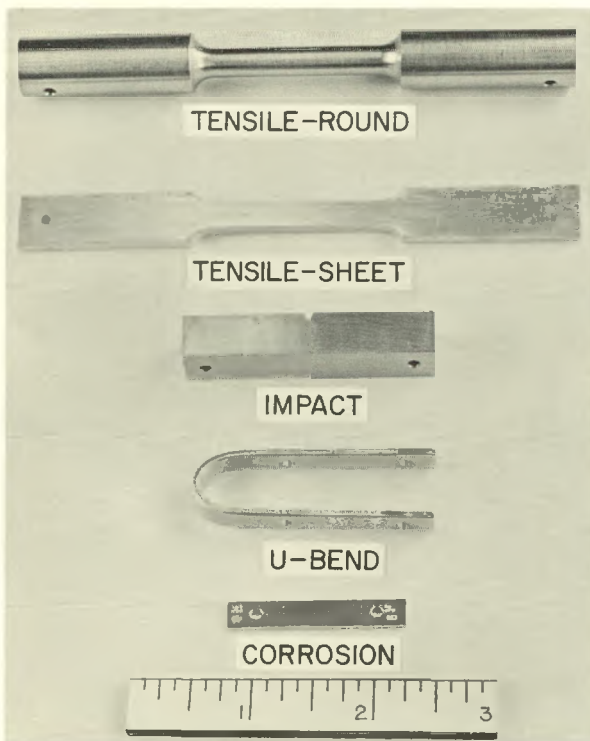


Figure 1. Typical Specimens Used in OMRE Corrosion Study

Corrosion specimens, 1 to 1-1/2 by 1/4 by 1/16 in., were machined on all surfaces from 1/8-in. sheet stock. Finishes were held to a specified 63 rms to ensure comparable surfaces for all specimens. Dissimilar metal specimens were obtained by butt welding two halves of these corrosion specimens. U-bend specimens, machined in a similar manner, were bent 180 degrees around a 1/4-in. -diam pin.

Round tensile specimens, having a 1/4-in. -diam test section, were used for the ferrous metals. The raw material for these specimens was in the form of 1-1/2-in. -diam bar stock. The bar was cut into 6-in. lengths and each length was quartered, so that four specimens were obtained per length. This procedure

minimized the total length of bar stock required; and, in addition, any variation in mechanical properties, due to inhomogeneity in the bar, was minimized.

Flat, 1-in. gauge length, tensile specimens with a 1/4 by 1/8 in. test section, were machined from aluminum and magnesium sheet stock. The longitudinal axis of the specimens was parallel to the rolling direction of the sheet.

TABLE I

CORROSION SPECIMENS IN THE OMRE

Material	Condition	Corrosion Specimens		Tensile Specimens		Impact Specimens		U - Bend Specimens	
		In Pile	Out of Pile	In Pile	Out of Pile	In Pile	Out of Pile	In Pile	Out of Pile
Type 1020 Carbon Steel	Normalized	6	7	4	3	4	5	4	1
Type 304 Stainless Steel	Annealed	7	7	4	3	5	5	4	1
Type 304 Stainless Steel	Sensitized 2 hr at 1200°F	7	8	3	3	5	4	4	1
Type 1100 Aluminum	Annealed	7	7	5	3	-	-	4	1
Magnesium	Annealed	7	7	4	3	-	-	-	-
Magnesium	Anodized	6	6	-	-	-	-	-	-
Type 410 Stainless Steel	Annealed	7	8	3	3	5	5	4	1
Type 4130 Alloy Steel	Normalized	7	8	4	3	5	5	4	1
Miscellaneous	Dissimilar welded Metals	6	2	-	-	-	-	-	-
Total		60	60	27	21	24	24	24	6





Standard V-notch Charpy impact specimens were machined from the 1-1/2-in. bar stock, by a procedure similar to that outlined for the round tensile specimens.

Representative samples of each of the stock materials were macroscopically and microscopically examined. Chemical analysis of the materials is given in Appendix B.

Immediately following machining, a thin coating of oil was applied to all specimen surfaces. After proper identification, by stamping, all dimensions were measured and recorded. Measurements were made to the nearest 0.001 in. A type QA Profilometer was employed to spot check the surface roughness of all finishes.

Degreasing was accomplished by a thorough rinsing in boiling trichloroethylene. All specimens were subsequently handled with white gloves and tweezers. The specimens were stored in a desiccator prior to loading.

C. SPECIMEN LOADING

In view of induced radioactivity, the postexposure examination of specimens contained in the dummy fuel element was conducted remotely. Consequently, specimen holders were designed for ease of disassembly (Figure 2).

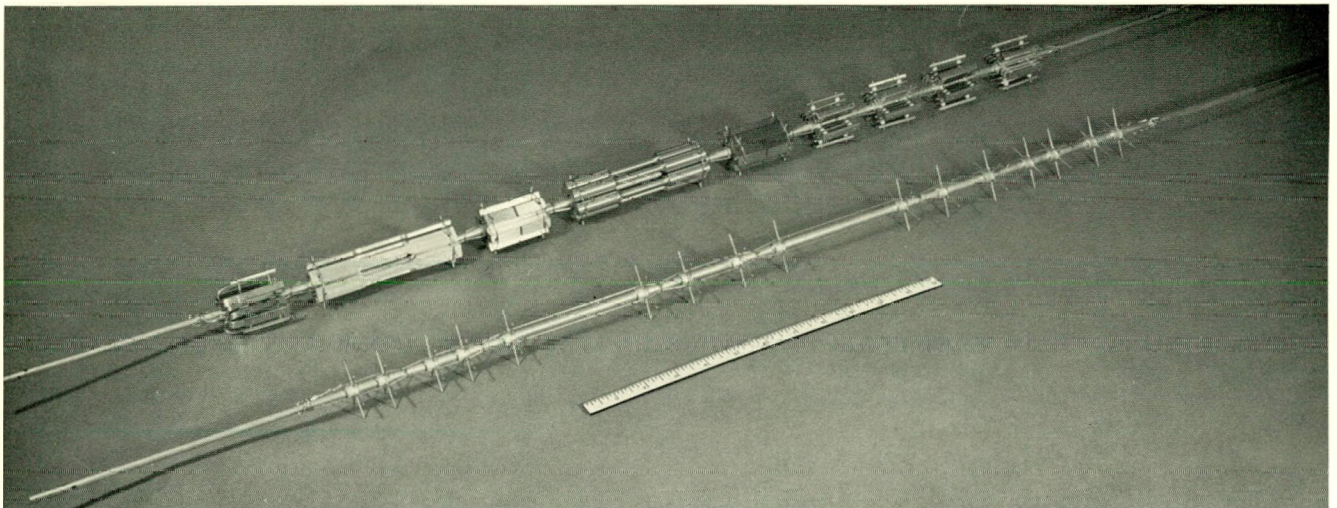


Figure 2. OMRE Dummy Element Specimen Holder

Specimen holders were welded assemblies, constructed to Type 304 stainless steel. Six-legged "spiders" were positioned and welded to a 1/4-in. center rod,

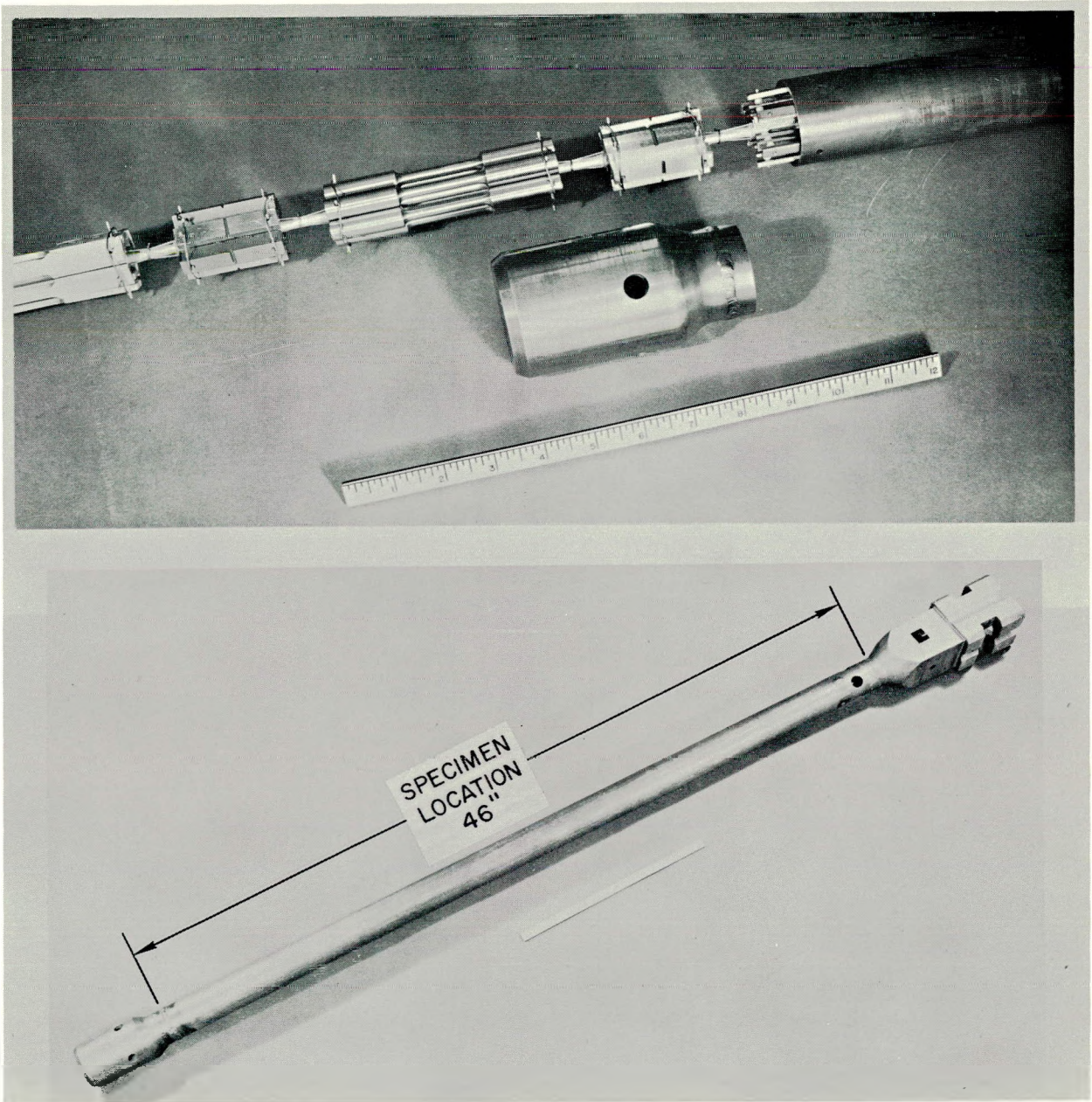


Figure 3. Inserting Specimens in an OMRE Dummy Element



for the proper alignment of specimens. Specimens were bundled along the center rod by individual mounting on the "spiders". Holes, $3/32$ in. in diameter, were drilled at both ends of each specimen for this purpose (see Figure 1). All corrosion specimens were rinsed in alcohol, dried, and weighed to the nearest 0.1 mgm, immediately prior to loading. Two dosimeter wires were strung along the center rods of each of the dummy element holders. A pure nickel wire (0.035-in. diam) was used for determining the fast flux, and an Al - 0.5 wt % Co wire (0.040-in. diam) for the thermal neutron flux.

The dummy elements provided a tubular length of 46 in., with a 1.87-in. ID for the placing of specimens (see Figure 3). The holder assemblies were fastened in the elements by means of two cross pins at each end of the holder. The ends of these pins were tack welded to the outer surface of the dummy elements (see Figure 3).

The loaded dummy elements were inserted in lattice positions 29 and 31 of the OMRE core (see Figure 4).

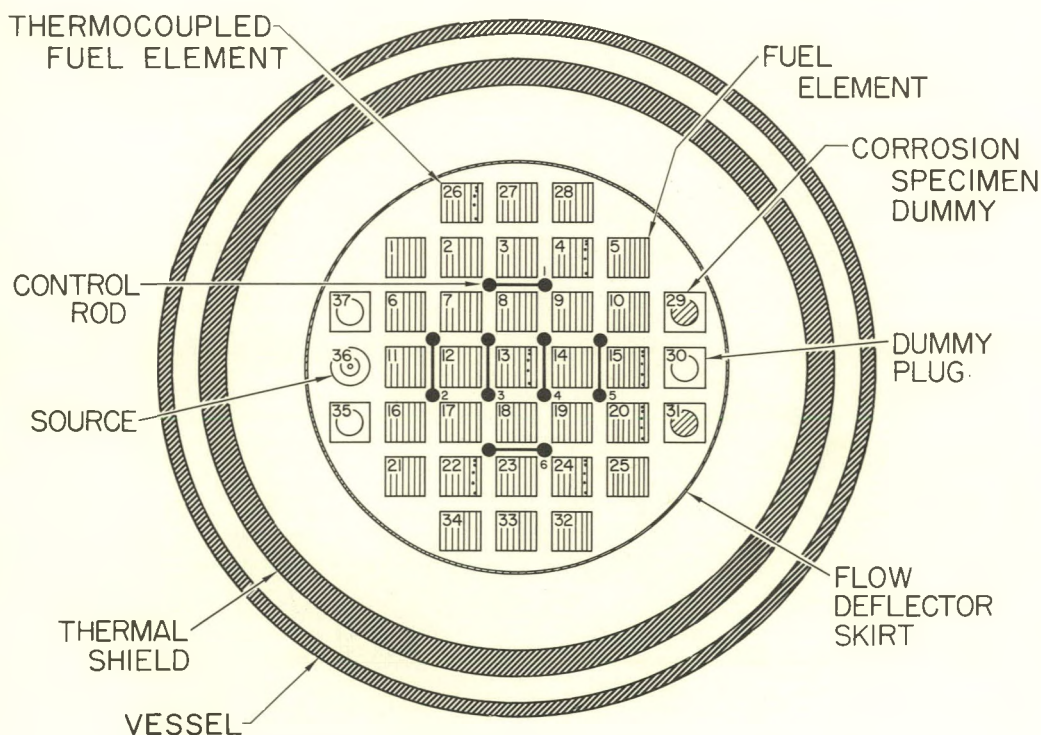


Figure 4. In-Core Location of Corrosion Study Elements



The out-of-pile specimens were located in a flanged spool of Schedule 40 two-inch carbon steel pipe. The specimen holder incorporated the same design principles used for the dummy element holders (see Figure 5). Specimens were inserted in the 47-in. horizontal length shown in Figure 6. Minor design modifications of the holder were made to accommodate the horizontal positioning. The spool was located in a circulating bypass line.

D. EXPOSURE CONDITIONS

Specimens contained in the dummy fuel element lattice position No. 29 were located in the OMRE core for eight months, during which the reactor produced 544 Mwd of thermal power. During the first three months of this exposure, the reactor was operated at various power levels and temperatures to determine reactor characteristics. Sustained power runs were realized for the remaining five months of the exposure. Figures 7 and 8 show nominal power levels and corresponding bulk coolant temperatures in the core which were achieved during the final five months of the dummy element exposure. Although the bulk coolant temperature reached as high as 700°F, it was held nominally at 600°F. Three major power level plateaus of 2, 4, and 6 Mwt are evident.

Santowax OM, a polyphenyl consisting of approximately 18% diphenyl, 47% ortho-terphenyl, 30% meta-terphenyl, and 5% para-terphenyl served as the moderator-coolant. These polyphenyls experience both radiolytic and pyrolytic decomposition under reactor conditions. A complex of isomers is so introduced. The major portion of these decomposition products, exclusive of the gases formed, is termed high boiler (more commonly referred to as HB). High boiler is defined as material less volatile than para-terphenyl.

The presence of HB effects a change in coolant properties, such as density, viscosity, and coefficient of thermal conductivity. The HB is removed by distillation. Hence, the amount of HB present is a function of temperature, intensity of radiation, total irradiation, and the rate of purification during reactor operation. The HB level during the exposure, expressed as weight percent, is shown in Figure 8. Although the HB content was varied over a wide range (0 to 41 wt %), the relative isomeric ratio of the unaffected coolant remained essentially that of the virgin coolant.

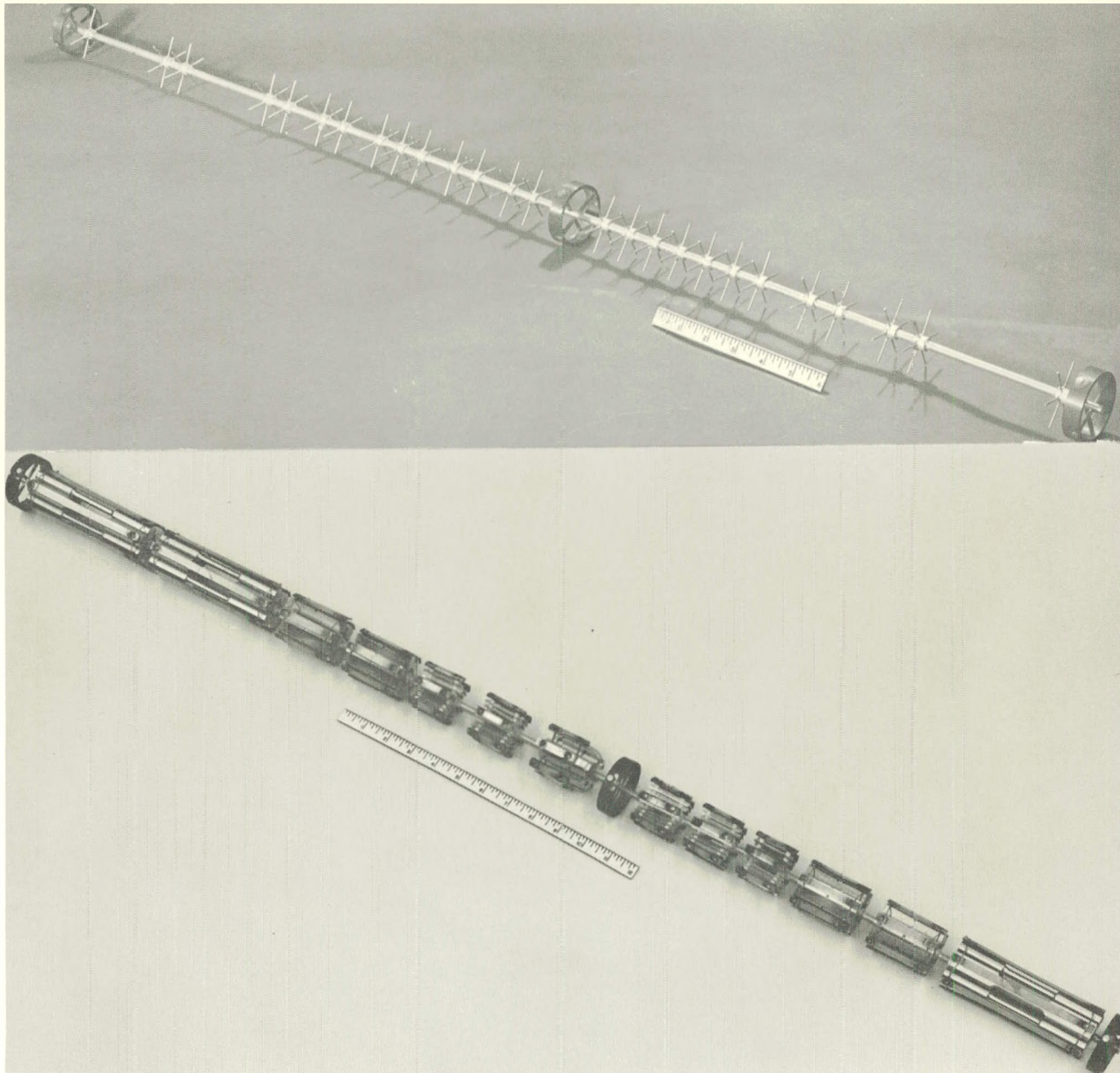


Figure 5. Bypass Specimen Holder Assembly



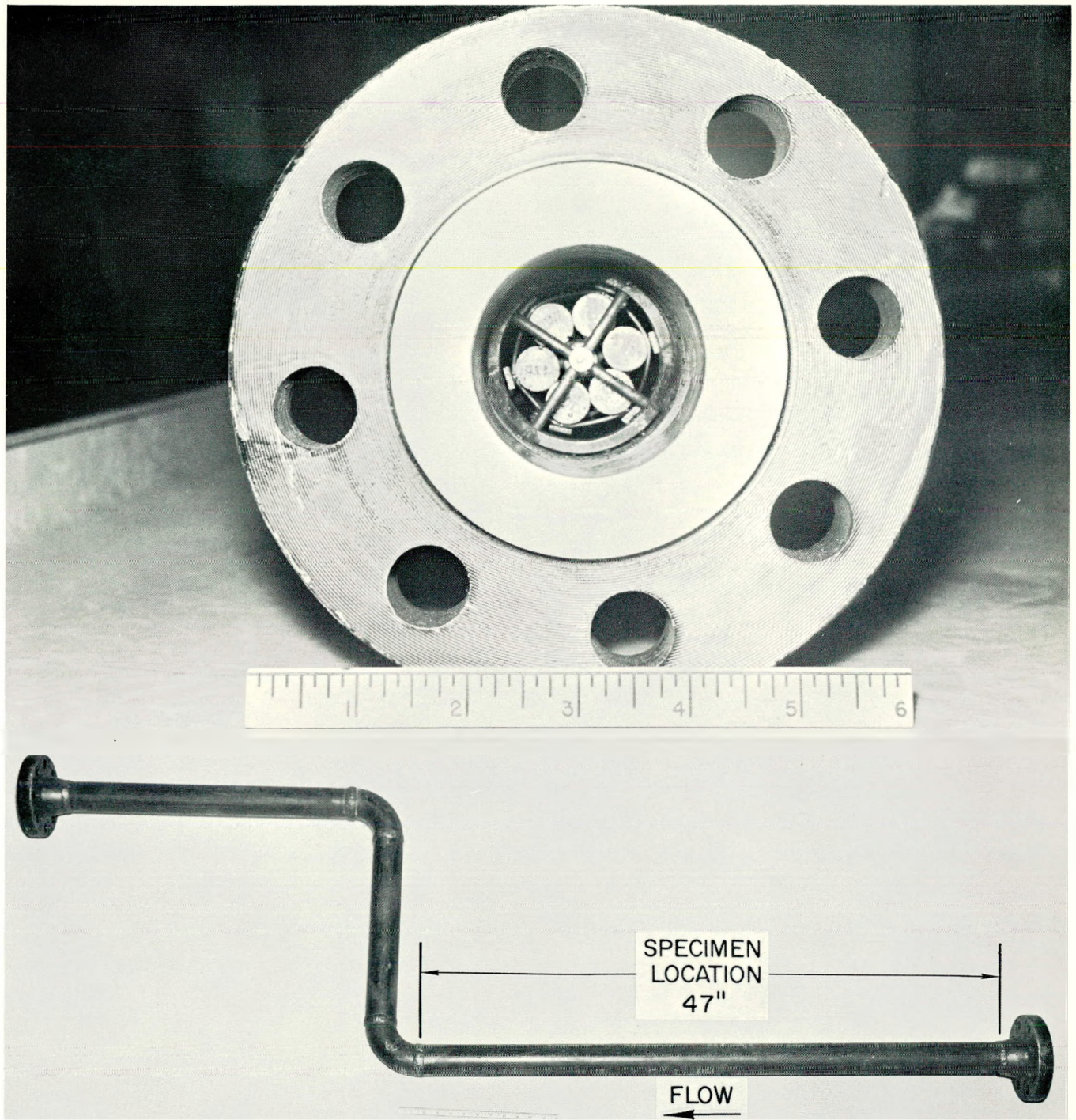


Figure 6. Location of Specimens in OMRE Bypass

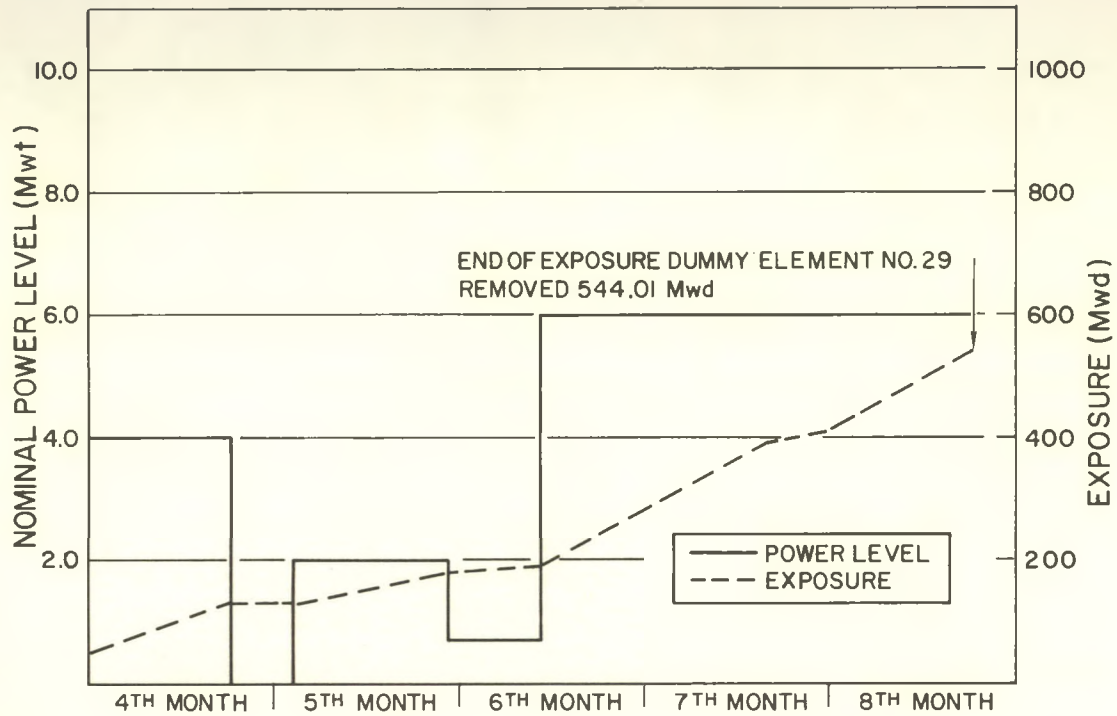


Figure 7. Exposure Conditions in the OMRE
(Power Level and Exposure)

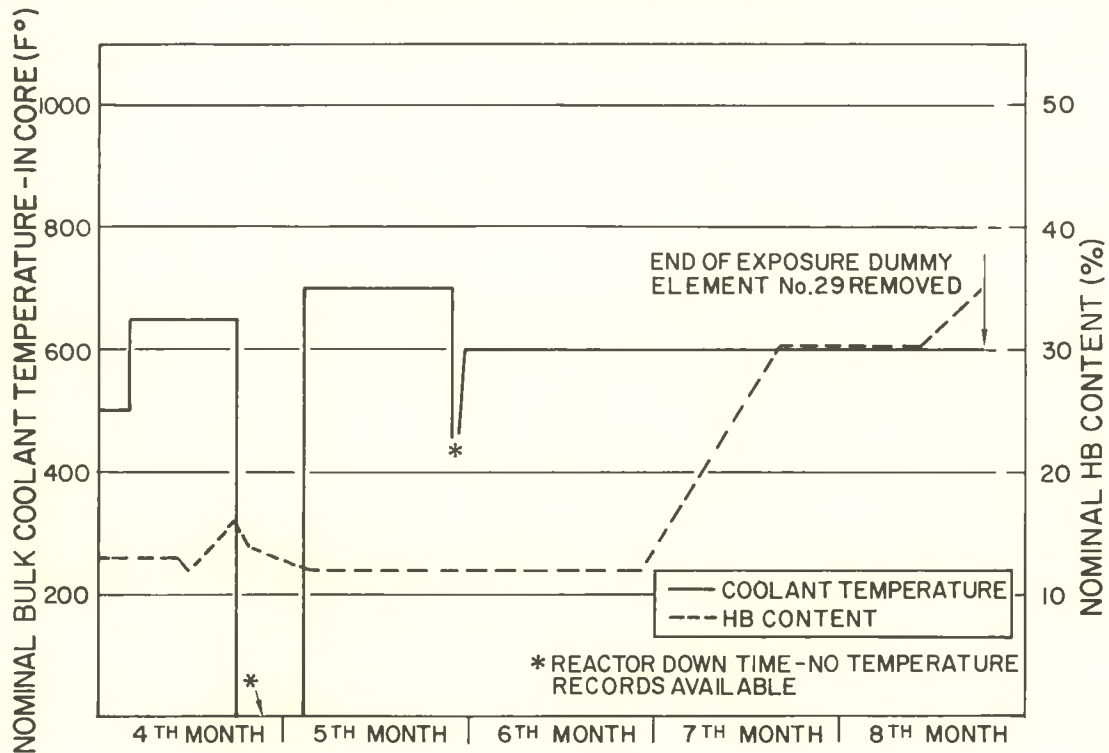


Figure 8. Exposure Conditions in the OMRE
(Bulk Coolant Temperature and HB Content)



The decomposition gases formed are principally hydrogen, ethane, methane, and propane. The system pressure (approximately 200 psi) was maintained with nitrogen.

Coolant samples were periodically taken, during reactor operation, for water determinations. The water content of 14 samples, determined by the Karl Fischer method, ranged from a minimum of 18 to a maximum of 720 ppm.

The out-of-pile specimens were inserted in the bypass line during the construction of the reactor. Consequently, the specimens were in line for 18 months, during which the reactor was in actual operation for only 13 months. Temperatures in the location of the specimens were estimated to be approximately 20°F below the bulk coolant temperature in the core. The thermal and other exposure history of the out-of-pile specimens may be taken from Figures 7 and 8 for the first 8 months of the exposure. During the remaining 5 months of the out-of-pile exposure, the reactor was operated at levels up to 10 Mwt. The bulk coolant temperature in the core was maintained at a nominal 600°F. HB content varied from 30 to 41 wt %.

Flow rates at both the in-pile and out-of-pile specimen locations were estimated to be 0.3 to 0.5 fps.

E. POSTEXPOSURE EXAMINATION

The corrosion study dummy fuel element, located in core position No. 29, was lifted from the core directly into a lead shipping cask. The disassembly of the element and removal of specimens was accomplished in hot cells operated by Battelle Memorial Institute.

The specimen-bearing holder was disengaged with relative ease. Saw cuts were made through the element, just inside the support pins at each end of the specimen holder. The specimen-bearing holder was removed and dipped in trichloroethylene. Figure 9 shows the holder, with specimens attached, shortly after its removal from the element. A rather thick carbonaceous deposit was evidenced on all surfaces of the holder and specimens. In some areas, the black deposit flaked and washed off to reveal shiny metallic surfaces. These areas were found primarily on Type 304 stainless steel surfaces. The specimens

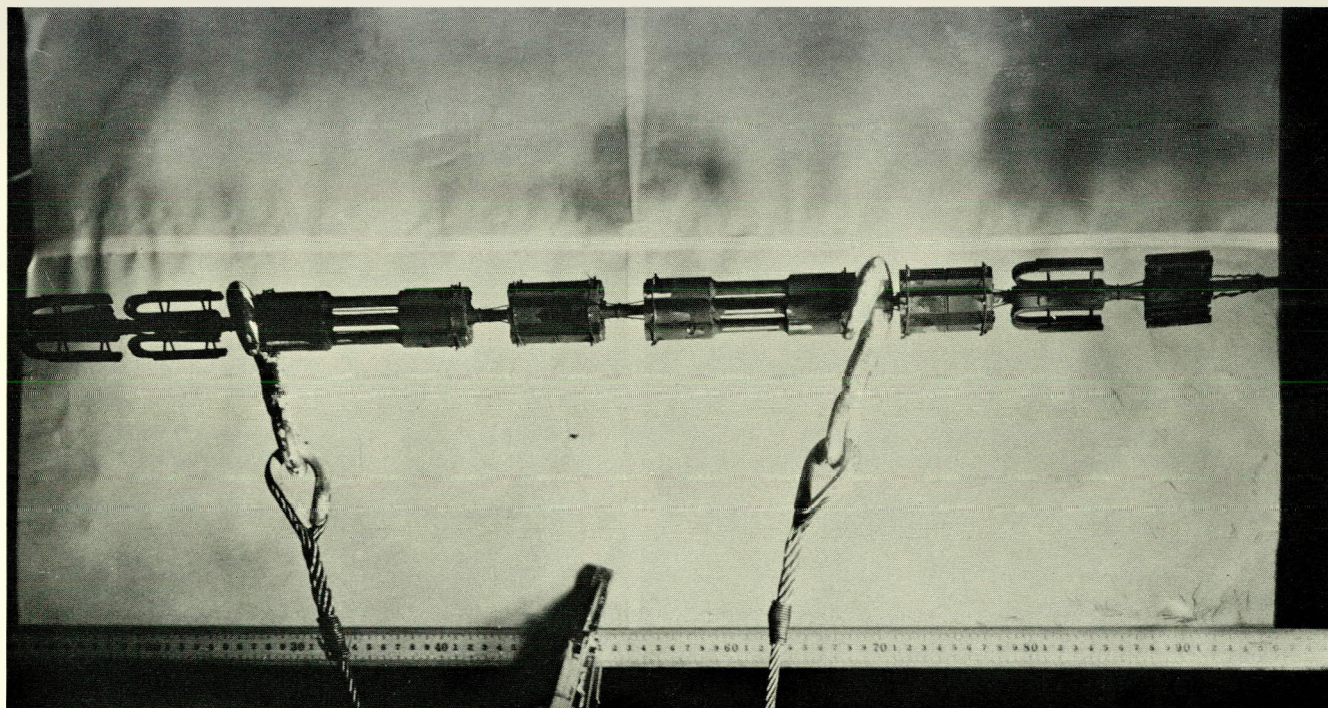


Figure 9. Specimen Holder Shortly After Removal From OMRE
Dummy Fuel Element

were removed from the holder and brushed individually in trichloroethylene to facilitate identification. The stamped markings were readily discernible.

The dosimeter wires strung along the center rod of the holder were recovered intact. The Al - 0.5 wt % Co wire was scanned, over its length, for relative gamma activity in the 1.10 to 1.35 Mev range. The relative gamma intensity curve is shown in Figure 10. The approximate specimen bundle locations are also shown. Three 2-in. sections of the aluminum-cobalt wire were removed for radiochemical analysis. The relation of thermal flux, as determined by radiochemical techniques and the relative gamma intensity scan, is shown in Figure 11.

Attempts to scan the nickel dosimeter wire for relative gamma activity were unsuccessful. The brittleness of the wire prevented its loading in the scanning apparatus. The integrated fast flux was determined by radiochemical analysis of six samples of the nickel wire.

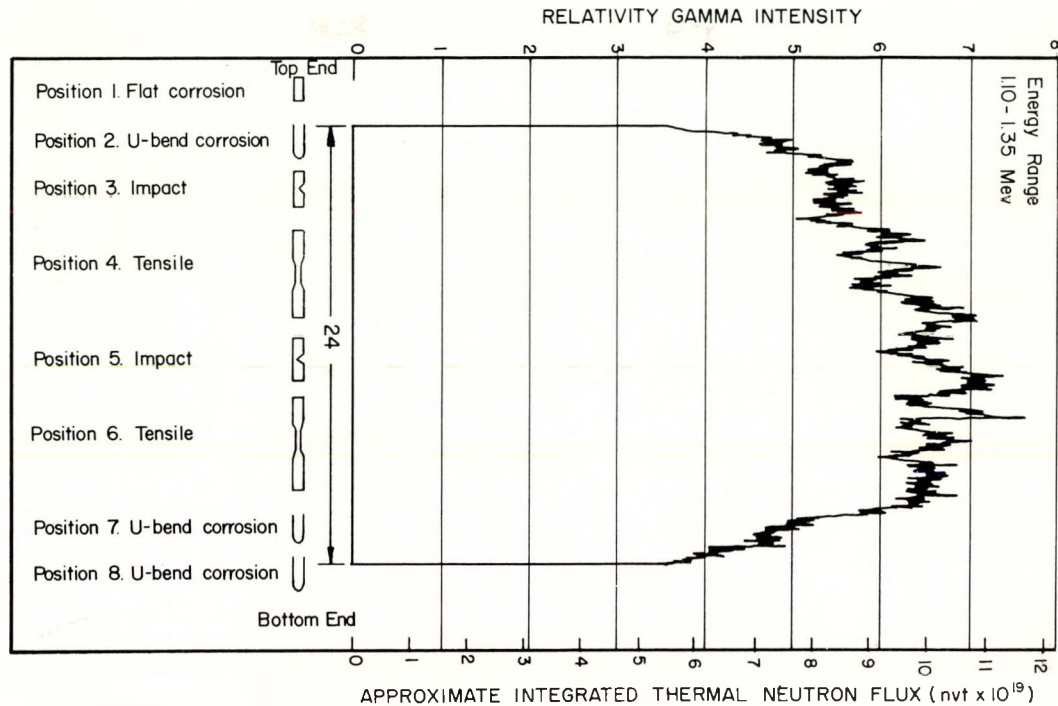


Figure 10. Relative Gamma Ray Intensity Curve of Aluminum - 0.5 Wt % Cobalt Dosimeter Wire

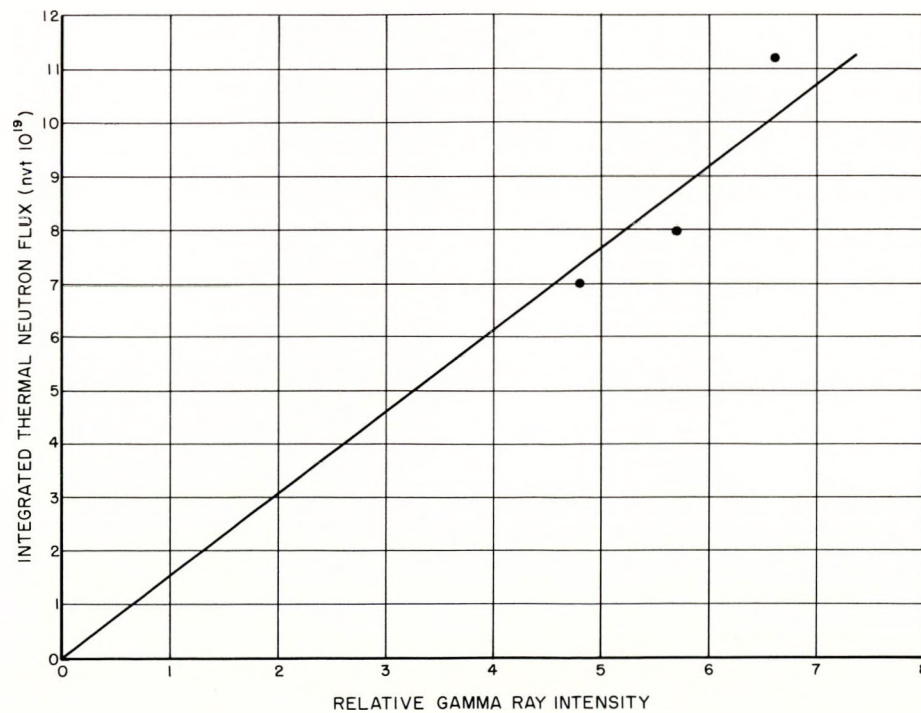


Figure 11. Relation of Integrated Flux as Determined From Dosimeter Analysis and the Relative Gamma Ray Intensity

POSITION NO.	TYPE OF SPECIMEN	NO. OF SPECIMENS	IDENTIFICATION NO.
1	CORROSION	12	B7K, C7K, D7K, E7K, J7K, H7K, A7K/C8K, A7K/B8K, A8K/B8K, C8K/J8K, H8K/J8K, A8K/H8K
2	U-BEND	6	J30K, J31K, J32K, B33K, C34K, C35K
3	IMPACT	6	B20K, B21K, B22K, B23K, C24K, C25K
4	TENSILE	6	B10K, B11K, B12K, C10K, C11K, C12K
5	IMPACT	6	H20K, H21K, H22K, H23K, C22K, C23K
6	TENSILE	6	H10K, H11K, H12K, A13K, B13K, J13K
7	U-BEND	6	B30K, B31K, B32K, C32K, C33K, C34K
8	U-BEND	6	A30K, A31K, A32K, H32K, H33K, H34K

THERMAL FLUX ($\times 10^{19}$ nvt)
(ALUMINUM-0.5 wt % COBALT WIRE)

FAST FLUX ($\times 10^{19}$ nvt)
(> 1 Mev)
(NICKEL WIRE)

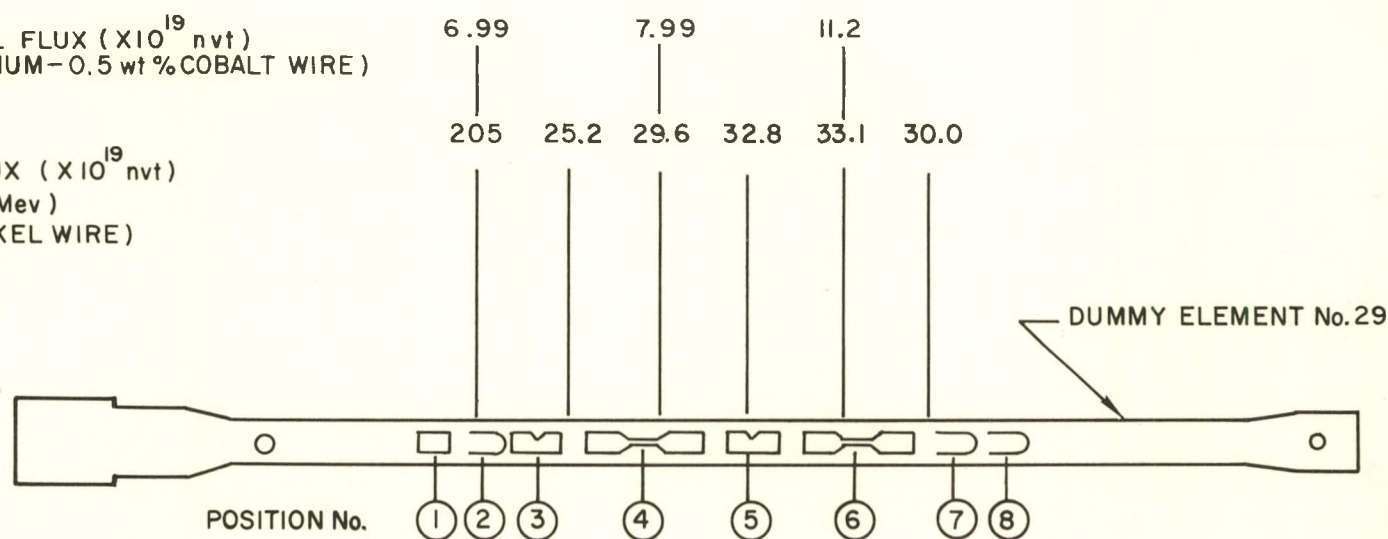
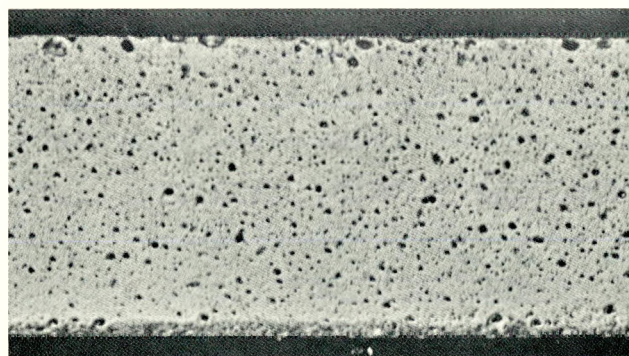


Figure 12. Neutron Flux Distribution Determined by Radiochemical Techniques

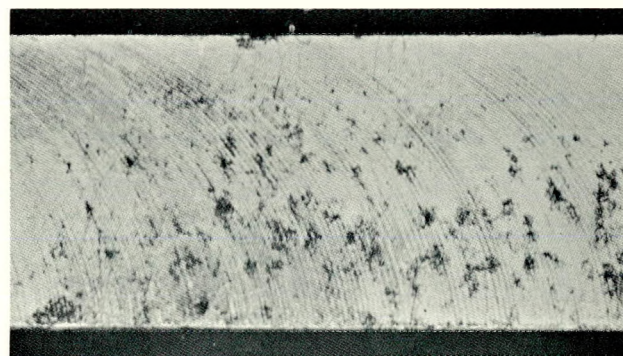


A summary of integrated thermal and fast neutron fluxes, as determined by radiochemical analysis, is shown in Figure 12. The relative location of dosimeter wire samples for analyses is also shown. The average thermal integrated neutron flux ranged from 6.99 to 11.2×10^{19} nvt. Average fast neutron flux ranged from 20.5 to 33.1×10^{19} nvt. All radiochemical analyses were performed by Battelle personnel. An explanation of flux determination techniques and sample calculations are found in Appendix A.

The corrosion and U-bend specimens were initially cleaned by soaking and brushing in trichloroethylene. This procedure was considered inadequate for removing all of the residual polyphenyl. The specimens were subsequently soaked in butyrolactone and Lestoil* and lightly buffed with a rotating fiber brush. The corrosion specimens were rinsed in carbon tetrachloride, dried, and weighed to the nearest 0.1 mgm. Unfortunately, the aluminum and magnesium specimens were chemically attacked by the Lestoil solution. Unexposed control specimens of these materials were cleaned in butyrolactone and Lestoil and examined. The aluminum control coupon was badly pitted by the Lestoil and experienced a significant weight loss. Although the magnesium control coupon did not experience a large weight loss when cleaned in Lestoil, pitting of the surface was apparent. The condition of these specimens after the cleaning test is shown in Figure 13.



Aluminum 6X



Magnesium 6X

Figure 13. Control Specimens after Cleaning in Lestoil

Weight change data obtained on the irradiated corrosion coupons are presented in Table II. The adverse effect of the cleaning solvent (Lestoil) on the aluminum and magnesium corrosion coupons rendered the weight change data

*Lestoil - trade name of a commercial solvent.



TABLE II

IN-PILE WEIGHT CHANGES

Material	Weight Change* per Unit Area (mgm/cm ²)
Type 304 Stainless Steel	+0.01
Type 304 Stainless Steel (sensitized 2 hr at 1200°F)	+0.01
Type 410 Stainless Steel	+0.85
Type 4130 Alloy Steel	-0.53
Aluminum	-2.95
Magnesium	-17.80
Aluminum† (control)	-2.61
Magnesium†	-0.18

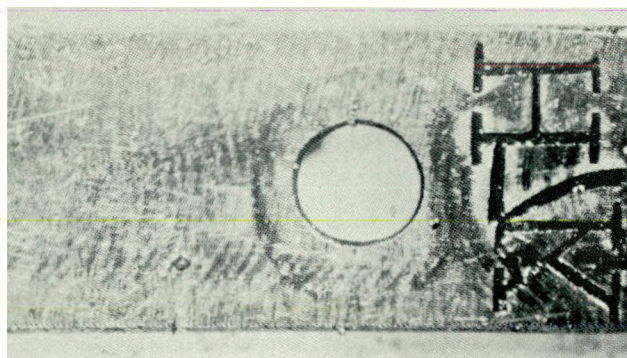
*Based on single value per material. Surface area of all specimens was 6.2 cm²

†Control specimens cleaned in Lestoil

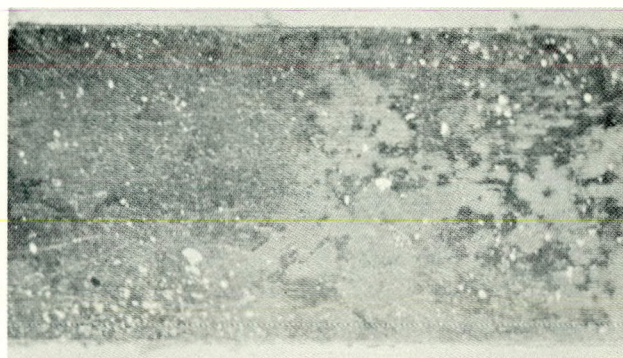
on these materials highly questionable. Table II includes data on the cleaning test as well as the postirradiation weight changes.

The Type 304 stainless steel specimens suffered the least attack. The weight gain recorded for the sensitized specimen, C7K, was in good agreement with that obtained on the annealed specimen, B23K. Slightly higher weight gains were obtained on the Type 410 stainless steel specimen. The Type 4130 steel coupon experienced a slight weight loss. All surfaces of the ferrous materials were uniform, with no evidence of localized attack. A thin dark film remained on the surface of the Type 4130 coupon. Surfaces of the Types 304 and 410 stainless steels were only slightly darkened during the exposure. Photographs of these specimens are found in Figure 14.

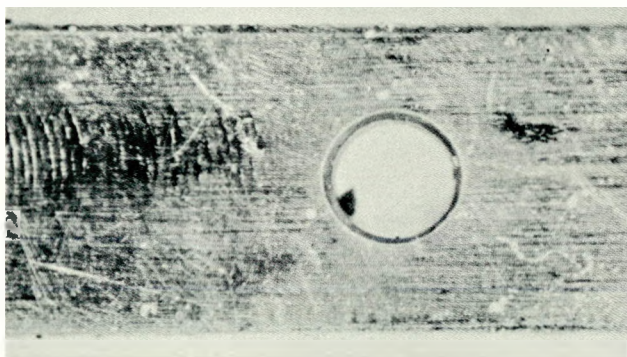
Six irradiated bimetal specimens were cleaned and visually examined. The bimetal couples included various combinations of Type 1020 carbon steel, Type 4130 alloy steel, and Types 304 and 410 stainless steels. No evidence of localized attack was found.



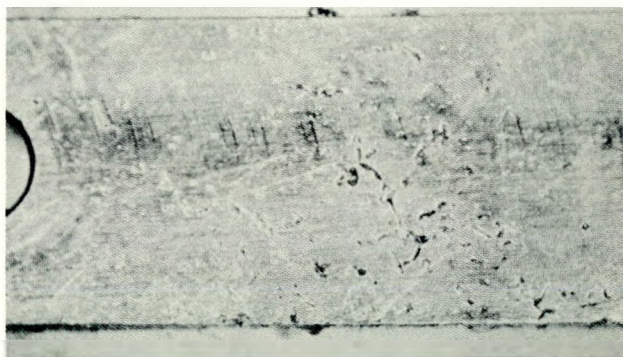
Type 410 Stainless Steel 6X



Type 4130 Steel 6X



Type 304 Stainless Steel 6X



Type 304 Stainless Steel 6X
(Sensitized)

Figure 14. Postexposure Appearance of In-Pile Corrosion Specimens



U-bend specimens included in the dummy element were also cleaned and visually examined. Indications of accelerated attack in the stressed areas of the bend were not found.

Tensile specimens irradiated in the dummy element were tested to failure. Control and furnace-treated specimens were also tested. As-machined specimens were used as controls. The furnace treatment was accomplished in a forced air convection furnace at 600°F for five months. All tensile testing was performed at room temperature.

Results of the tensile tests are reported in Table B-II of Appendix B. An approximation of radiation damage effects is made by comparing results obtained on the furnace-treated specimens with those obtained on the in-pile specimens. The ferritic steels (Type 410, and AISI 4130 and 1020) showed significant increase in ultimate tensile strength and in yield strength after exposure to irradiation. These increases were accompanied by a marked decrease in percent reduction in area and, to a lesser degree, in percent elongation. Similar trends, although generally of smaller magnitude, were noted for the irradiated Type 304 stainless steel specimens. Metallographic specimens of the Type 410 stainless steel and Type 1020 steel were prepared. No alteration of the microstructures was noted.

Postirradiation V-notch Charpy impact values are reported in Table B-III of Appendix B. Control, out-of-pile, and furnace-treated specimen results are also included for comparison. Impact specimens of AISI 1020 and 4130 steel were not included in the dummy element examined. Type 410 stainless steel experienced a significant reduction in impact strength, whereas the Type 304 stainless steel impact properties were not noticeably affected by irradiation, under the conditions of the test. Impact tests were conducted at room temperature. The impact values, as reported, are based on a maximum deliverable energy of 120 ft-lb.

The out-of-pile section of the bypass line containing the corrosion study specimens was removed for specimen examination. The pipe spool was completely drained of residual liquid polyphenyl coolant before shipment. The end flanges were sealed and the spool was crated for shipment. No auxiliary shielding of the wooden shipping crate was necessary. With the exception of initial decontamination, all handling and testing of the contained specimens was performed under normal laboratory techniques.



When removed, all surfaces of the out-of-pile specimen holder assembly and the specimens had a dark, carbonaceous deposit. The general appearance of the assembly, at this point, was quite similar to that observed in the examination of the dummy element (see Figure 15). Decontamination was accomplished by brushing each specimen in trichloroethylene. The black, contaminated residue was readily removed by this process.

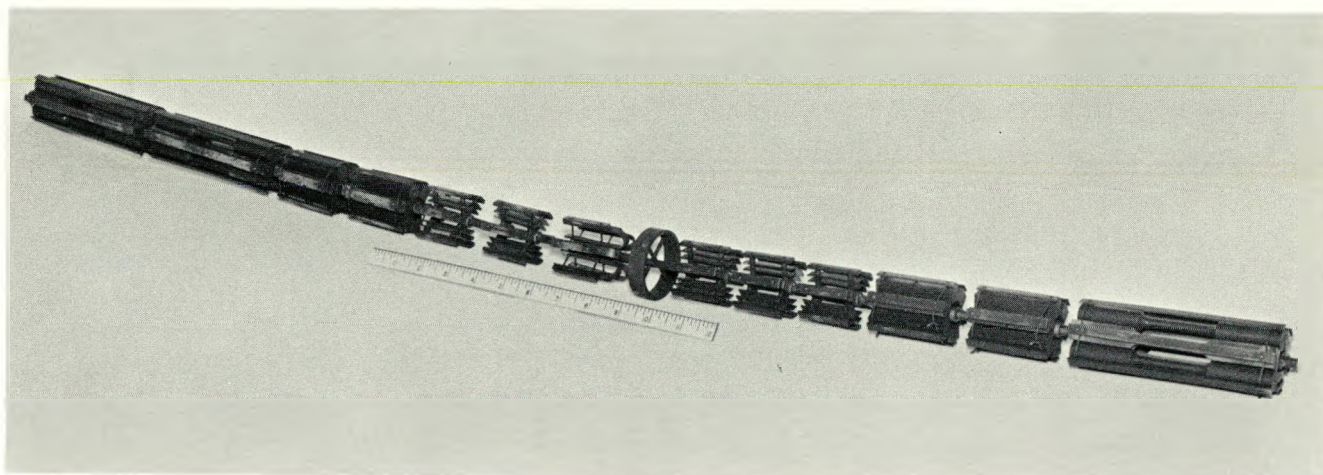


Figure 15. Bypass Specimen Holder Assembly after Removal

Visual examination indicated that the stainless steel (Types 304 and 410) and aluminum surfaces were in excellent condition. The AISI 4130 and 1020 steel specimen surfaces retained a dull, uniform, gray-to-black film. Magnesium surfaces exhibited pitting and general roughening. Anodized magnesium coatings were badly spalled and the bared areas exhibited serious pitting and general attack (see Figure 16).

The corrosion coupons were rinsed in acetone and alcohol, dried, and weighed to the nearest 0.1 mgm. Weight change data for the corrosion coupons and bimetal couples, exposed in the out-of-pile bypass line are presented in Table III. Weight change of the bimetal specimens did not indicate any accelerated attack.

The U-bend specimens were thoroughly cleaned and the stressed areas visually examined. No abnormalities were revealed (see Figure 17).

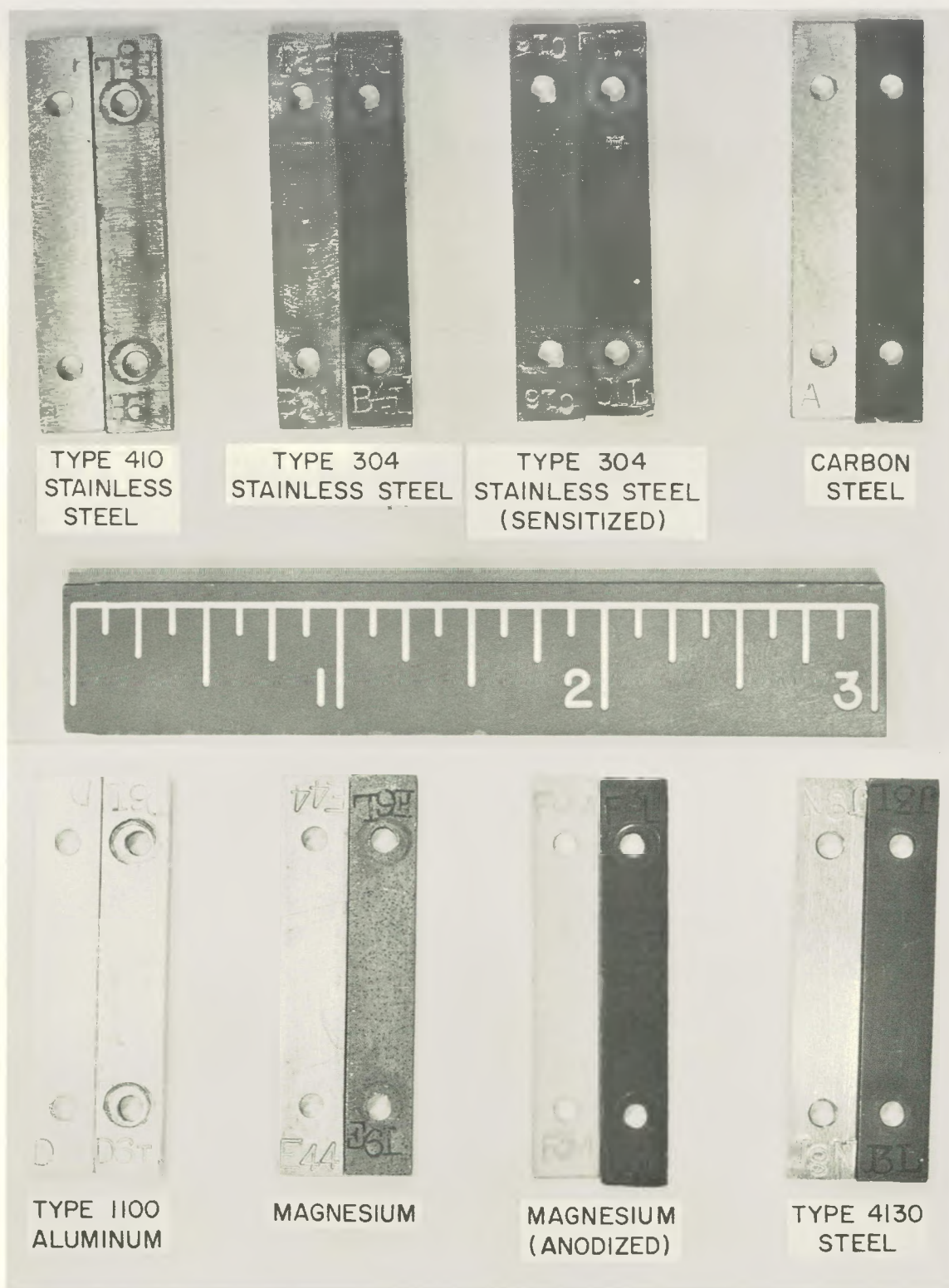


Figure 16. Corrosion Specimens Exposed in OMRE Bypass Line
(Note: Specimens on left are unexposed control specimens for comparison)



TABLE III

OUT-OF-PILE WEIGHT CHANGES

Material	Average Weight Change per Unit Area (mgm/cm ²) *
Type 1020 Carbon Steel	-0.3
Type 4130 Alloy Steel	-0.4
Type 410 Stainless Steel	-0.1
Type 304 Stainless Steel	+0.0
Type 304 Stainless Steel (sensitized 2 hr at 1200°F)	-0.0
Aluminum	-0.4
Magnesium	-9.5
Anodized Magnesium (heavy)	-3.2 †
Anodized Magnesium (light)	+1.1 †
304SS-1020 Carbon Steel (welded couple)	-0.2 §

*Based on algebraic average of 7 to 8 specimens, except where noted
Surface area of all specimens was 6.2 cm²

†Based on 3 specimens

§Based on 2 specimens

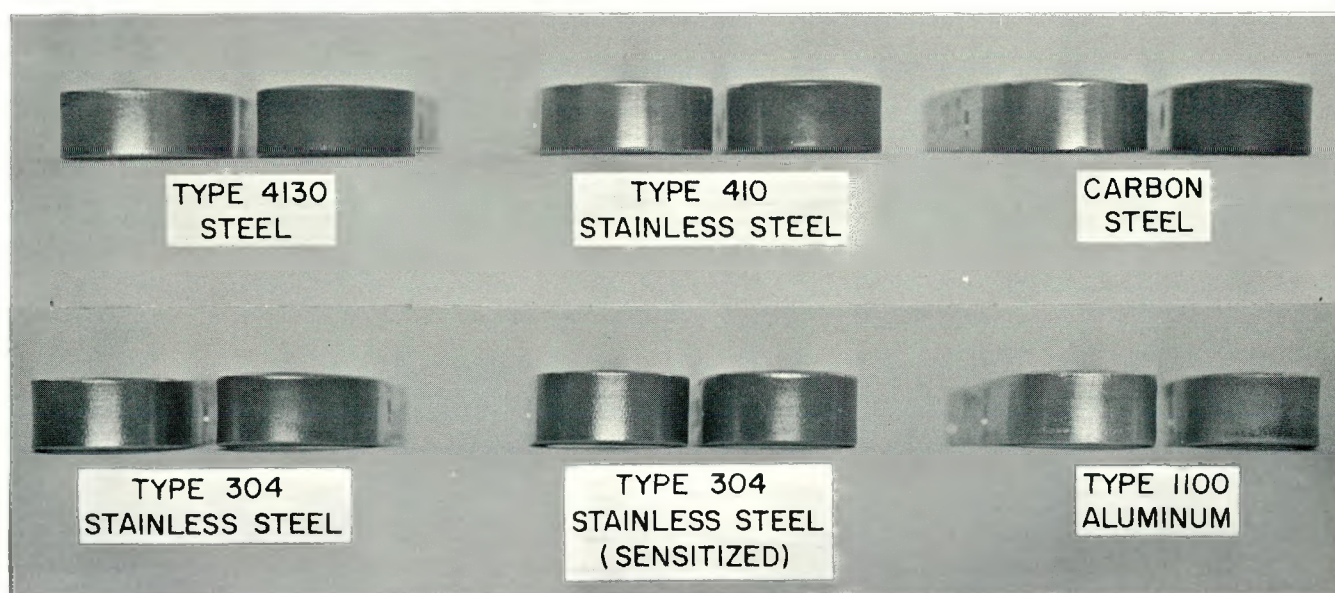


Figure 17. U-Bend Specimens Exposed in the OMRE Bypass Line
(Note: Specimens on left are unexposed control specimens for comparison)



The subsized tensile specimens and standard V-notch Charpy impact specimens exposed in the bypass line were tested to failure. Postexposure mechanical properties are tabulated in Tables B-II and B-III in Appendix B. These data agree well with those obtained after the five-month furnace treatment at 600°F.

TABLE IV
OMRE EXPOSURE
SUMMARY OF ROOM TEMPERATURE TENSILE PROPERTIES

Material*	Ultimate Strength (psi) (x 10 ⁻³)			Yield Strength (psi) (x 10 ⁻³)			Reduction in Area (%)			Elongation (% in 1 in.)		
	Furnace [†] Treated at 600°F	Out [§] of Pile	In Pile **	Furnace [†] Treated at 600°F	Out [§] of Pile	In Pile **	Furnace [†] Treated at 600°F	Out [§] of Pile	In Pile **	Furnace [†] Treated at 600°F	Out [§] of Pile	In Pile **
Type 1020 Carbon Steel	67.2	67.3	78.4	46.3	41.1	58.5	52.3	58.4	36.5	33.7	29.5	28.4
Type 304 Stainless Steel (annealed)	85.4	85.9	83.9	39.6	42.0	47.2	75.7	75.0	55.9	69.2	70.0	63.2
Type 304 Stainless Steel (sensitized for 2 hr at 1200°F)	85.1	85.3	86.1	45.0	40.9	49.9	74.3	76.2	53.5	63.2	67.7	59.5
Type 4130 Alloy Steel	103.3	104.4	109.0	73.2	73.2	80.0	55.7	53.8	48.4	26.7	24.3	31.0
Type 410 Stainless Steel	93.0	92.4	102.8	69.6	70.0	86.3	74.0	73.8	44.3	32.7	31.7	25.4
Type 1100-0 Aluminum	11.5	11.4	-	4.7	4.4	-	-	-	-	-	-	-
Magnesium	31.6	29.0	-	13.0	15.7	-	-	-	-	-	-	-

*Average values reported, based on 1 to 4 specimens per material

†Furnace treated in open atmosphere for 5 months at 600°F

§Specimens exposed in OMRE bypass line for 13 months at a nominal temperature of 580°F

**Specimens exposed in OMRE dummy element #29 for 8 months at a nominal temperature of 600°F





obtained after the furnace treatment at 600°F. Property changes observed for the out-of-pile specimens were concluded to be thermally induced.

TABLE V

OMRE EXPOSURE

SUMMARY OF ROOM TEMPERATURE IMPACT PROPERTIES

Material	Impact Strength (ft-lb)*		
	Furnace Treated [†] at 600°F	Out of Pile [§]	In Pile ^{**}
Type 1020 Carbon Steel	88.3	88.1	-
Type 304 Stainless Steel	120+	120+	120+
Type 304 Stainless Steel (sensitized 2 hr at 1200°F)	120+	120+	120+
Type 410 Stainless Steel	120+	120+	88.7
Type 4130 Alloy Steel	63.3	66.0	-

*Average values reported, based on 3 to 5 specimens per material

†Furnace treated in open atmosphere for 5 months at 600°F

§Specimens exposed in OMRE bypass line for 13 months at a nominal temperature of 580°F

**Specimens exposed in OMRE dummy element #29 for 8 months at a nominal temperature of 600°F

Mechanical property changes experienced by the in-pile specimens were no greater than the change expected of irradiated specimens without the polyphenyl environment.⁶ An increase in ultimate and yield strengths was accompanied by a decrease in percent reduction in area and elongation. Yield strength and percent reduction in area were the mechanical properties most sensitive to radiation damage effects. Austenitic stainless steel specimens appeared most resistant to radiation damage. Impact values for specimens exposed in pile were obtained on Types 304 and 410 stainless steel. Impact properties of the Type 304 were apparently unaffected, whereas those of the Type 410 were significantly reduced under conditions of the test.



IV. CONCLUSIONS

The polyphenyl environment of an organic moderated and cooled reactor is relatively noncorrosive to most structural and cladding materials. The behavior of Types 304 and 410 stainless steel is considered excellent. The attack on aluminum, Type 4130 alloy steel, and Type 1020 carbon steel is very mild. Gross pitting and general attack was experienced by the magnesium specimens exposed. Anodized surfaces did reduce weight changes for magnesium; however, undesirable localized pitting of the magnesium prevailed.

Irradiation in the order of 7 to 11×10^{19} nvt thermal and 20 to 33×10^{19} nvt fast apparently did not seriously affect the corrosion resistance of the materials tested. The environment produced neither stress corrosion nor galvanic attack.

Mechanical property changes after irradiation were unaffected by the polyphenyl environment. These changes were the same as those expected in materials irradiated without a polyphenyl environment. These changes were most predominant in the ferritic grade steels, as opposed to austenitic grades. Although embrittling tendencies were indicated, the degree of embrittlement was not serious. The microstructure of these materials was unaffected by irradiation. Specimens exposed in a nonirradiating environment in the bypass line experienced no mechanical property changes other than those induced thermally.



APPENDIX A

TECHNIQUES OF NEUTRON DOSIMETRY ANALYSIS

Sections cut from two neutron dosimeter wires irradiated by Atomics International in dummy fuel element OMR-1 were analyzed by radiochemical techniques to obtain flux data. A nickel wire was used to indicate the fast neutron flux and an aluminum - 0.5 wt % cobalt wire was used to measure the thermal neutron flux.

The procedure used to analyze one of the sections cut from the nickel dosimeter wire is explained below in detail. Analysis of the aluminum - 0.5 wt % cobalt was similar.

The wire, which was observed to have a black coating, was washed in a solution of nitric and hydrochloric acids, followed by water and acetone rinses. The wire was then dried and weighed.

After weighing, the wire was placed in a 25-ml volumetric flask and dissolved in a solution of nitric and hydrochloric acids. After the solution was complete, the liquid in the flask was diluted to volume and thoroughly mixed. Two 250-lambda aliquots were withdrawn from the flask and placed in separate 10-ml volumetric flasks. After filling the flasks to volume with acid, 1-ml aliquots were removed from the flasks for counting.

The 1-ml sample was then placed in a gamma ray spectrometer and the gamma spectrum was obtained in the energy range of 0 to 2 Mev. It was observed that gamma rays were present having energies corresponding to those of both cobalt-58 and cobalt-60. This necessitated correction of the observed counting rates for the presence of cobalt-60. The correction was made by counting the final aliquots above 1.0 and above 0.6 Mev and comparing the counts with cobalt-58 and cobalt-60 standards counted at the same energy levels. The cobalt-60 standard was obtained from Radiation Counter Laboratories, Skokie, Illinois, and found to be accurate by cross check with NBS and BMI 4 Pi standards. The cobalt-58 standard was prepared by 4 Pi counting, assuming a decay scheme with 85% electron capture, 15% positron emission, and a half-life of 72 days. The value for this cobalt-58 standard was 0.247 μ c on the day of counting.



The following table includes the count rates observed for a 1-ml aliquot of sample OMR-1-Ni-DW-1.

OBSERVED COUNT RATES

	Counts Per Minute Above 1 Mev	Counts Per Minute Above 0.6 Mev
Sample	82,887	231,961
Co ⁵⁸ Standard	4,647	41,327
Co ⁶⁰ Standard	248,521	414,063

From the above counting rates, the fractions of counts from the cobalt-58 and cobalt-60 standards occurring above 1 Mev, compared to those above 0.6 Mev, were calculated to be 0.112 for cobalt-58 and 0.601 for cobalt-60. Simultaneous equations were set up, using A as the cobalt-58 contribution to the total count rate above 0.6 Mev and B as the cobalt-60 contribution above 0.6 Mev. These equations were:

$$0.112 A + 0.601 B = 82,887 \text{ cpm}$$

$$A + B = 231,961 \text{ cpm};$$

therefore

$$0.112 A + 0.601(231,961 - A) = 82,887.$$

Solving for A:

$$A = 115,900 \text{ cpm.}$$

By comparing the cobalt-58 activity above 0.6 Mev of the sample with that of the standard, the activity of the sample in microcuries was determined

$$\frac{115,900 \text{ cpm}}{41,328 \text{ cpm}} \times 0.247 \mu\text{c} = 0.693 \mu\text{c.}$$



An average activity of the sample was determined from the above calculations and those of a second aliquot. The calculated sample activity was then determined in disintegrations per second-gram, for use in flux calculations:

$$D = \frac{(0.684 \mu\text{c/sample}) (10^3 \text{ dilution}) (3.7 \times 10^4 \text{ d/sec-}\mu\text{c})}{0.1040 \text{ gm/sample}}$$

$$= 2.43 \times 10^8 \text{ d/sec-gm,}$$

where

D = Disintegrations per second-gram (d/sec-gm).

The following equation was used for the flux calculation:

$$nv = \frac{D}{N \sigma \left(1 - e^{-\lambda t_1}\right) \left(e^{-\lambda t_2}\right)},$$

where

nv = Neutron flux (n/cm²-sec)

N = Number of atoms Ni per gram
 $= 6.98 \times 10^{21} \text{ atoms/gm (68\% Ni}^{58}\text{)}$

σ = cross section (cm²)
 $= 9.1 \times 10^{-26} \text{ cm}^2$

$1 - e^{-\lambda t_1}$ = Saturation factor for a reported irradiated time of 233 days
 $= 0.897$

$e^{-\lambda t_2}$ = Decay factor for 150 days decay time
 $= 0.232$

$$nv = \frac{2.43 \times 10^8 \text{ d/sec-gm}}{(6.98 \times 10^{21} \text{ atoms/gm}) (9.1 \times 10^{-26} \text{ cm}^2) (0.897) (0.232)}$$

$$= 1.84 \times 10^{12} \text{ n/cm}^2\text{-sec.}$$



In previous work, where the fast neutron flux obtained through cobalt-58 was compared with that obtained with P^{32} , it was found that the neutron threshold energy for cobalt-58 was 3.2 Mev. In order to obtain the flux over the whole fission neutron energy range, the calculated flux above was corrected:

$$\begin{aligned}nv &= \frac{1.84 \times 10^{12} \text{ n/cm}^2\text{-sec}}{0.18 \text{ (fraction of neutrons occurring above 3.2 Mev)}} \\&= 1.02 \times 10^{13} \text{ n/cm}^2\text{-sec.}\end{aligned}$$

The neutron flux values of the aluminum - 0.5 wt % cobalt dosimeter wires were determined by comparing the count rates of the samples with that of a cobalt-60 standard. The activity of the samples in disintegrations per second-gram were put into the same flux equation in determining the cobalt-58 flux values.

$$nv = \frac{\text{d/sec-gm}}{(1.02 \times 10^{22} \text{ atoms/gm}) (3.7 \times 10^{-23} \text{ cm}^2/\text{atom}) \left(1 - e^{-\lambda t_1}\right) \left(e^{-\lambda t_2}\right)}$$



APPENDIX B
CORROSION AND MECHANICAL
PROPERTIES OF MATERIALS EXPOSED
IN THE OMRE

TABLE B-II

CORROSION STUDY TENSILE DATA

Material	Ultimate Strength (psi) ($\times 10^{-3}$)				Yield Strength 0.2% Offset (psi) ($\times 10^{-3}$)				Elongation (% in 1 in.)				Reduction in Area (%)			
	Control *	Furnace Treated at 600°F †	Out of Pile §	In Pile **	Control *	Furnace Treated at 600°F †	Out of Pile §	In Pile **	Control *	Furnace Treated at 600°F †	Out of Pile §	In Pile **	Control *	Furnace Treated at 600°F †	Out of Pile §	In Pile **
Type 1020 Carbon Steel	65.3	67.2	67.7	78.4	43.3	48.6	40.6	58.5	-	25.5	30.0	28.4	65.3	47.0	60.5	36.5
	66.2	67.8	67.2	-	42.1	46.4	42.2	-	31.0	37.0	23.0	-	61.3	52.0	55.5	-
	65.2	66.5	66.9	-	40.5	43.8	41.4	-	25.0	39.0	35.5	-	59.5	58.0	59.1	-
Type 304 Stainless Steel (annealed)	85.1	84.8	84.6	83.7	40.4	40.0	41.7	43.0	72.0	67.5	72.0	61.0	78.9	75.0	77.2	56.5
	87.0	85.3	86.7	81.0	40.8	40.7	42.1	49.5	69.0	67.0	69.0	-	79.5	76.0	74.3	52.0
	85.0	86.0	86.3	84.8	41.8	38.1	42.1	49.0	78.0	73.0	69.0	62.5	84.7	76.0	73.6	52.2
				86.0				45.0				66.0				63.0
Type 304 Stainless Steel (sensitized for 2 hr at 1200°F)	85.3	83.4	85.5	84.4	40.2	39.0	41.1	49.2	70.0	67.0	67.0	-	75.4	75.0	77.2	47.2
	85.3	85.0	84.8	87.0	39.1	41.2	39.7	46.0	69.0	67.5	67.0	64.0	75.4	73.0	76.6	53.3
	86.3	87.0	85.5	87.7	39.8	54.8	42.0	54.5	70.0	55.0	69.0	55.0	78.5	75.0	74.8	60.0
Type 410 Stainless Steel	89.4	93.4	92.3	103.2	73.4	70.6	69.7	87.0	28.0	31.0	31.0	12.0	73.8	73.0	73.6	43.0
	89.1	92.8	92.6	102.5	75.3	68.7	70.1	85.9	26.0	34.0	34.0	26.3	73.3	75.0	74.2	34.0
	88.2	92.7	92.2	102.8	80.0	69.6	70.1	86.0	26.0	33.0	30.0	25.0	71.4	74.0	73.6	59.0
Type 4130 Alloy Steel	96.4	103.8	102.8	109.0	70.0	73.6	72.4	80.0	21.0	25.0	23.0	31.0	57.0	55.0	56.2	48.4
	95.5	104.4	100.1		66.7	76.9	74.3		25.0	-	25.5	-	61.0	56.0	54.0	
	98.4	101.7	105.0		76.0	69.2	72.8		-	28.5	25.0		57.7	56.0	51.2	
Type 1100-0 Aluminum	12.0	11.3	11.5		5.7	3.8	4.5									
	12.1	11.3	11.3		5.5	4.8	4.7									
	12.0	11.8	11.3		4.8	4.5	4.0									
Magnesium	32.4	31.4	29.2		25.4	17.1	15.8									
	32.4	31.8	29.0		23.7	7.7	15.4									
	31.1	31.6	28.9		22.1	14.2	16.0									

*Values obtained on as-machined specimens

†Furnace treated in open atmosphere for 5 months at 600°F

§Specimens in OMRE bypass line for 13 months at a nominal temperature of 580°F

**Specimens in OMRE dummy element #29 for 8 months at a nominal temperature of 600°F





TABLE B-III

CORROSION STUDY IMPACT DATA

Material	Charpy V-Notch Impact Values (ft-lb)*			
	Control [†]	Furnace [§] Treated At 600°F	Out of Pile ^{**}	In Pile ^{††}
Type 1020 Steel	69.0	93.5	86.0	-
	65.0	91.5	85.7	-
	64.0	80.0	92.5	-
			102.3	
Type 304 Stainless Steel	120+	120+	120+	120+
	120+	120+	120+	120+
	120+	120+	120+	120+
Type 304 Stainless Steel (sensitized for 2 hr at 1200°F)	120+	120+	120+	120+
	120+	120+	120+	120+
	120+	120+	120+	120+
Type 410 Stainless Steel	120+	120+	120+	77.0
	120+	120+	120+	93.4
	120+	120+	120+	95.2
				89.1
Type 4130 Alloy Steel	58.5	40.0	55.5	-
	40.0	80.5	52.0	-
	50.0	69.5	73.0	-
			65.0	-
			72.5	-

*Based on maximum deliverable energy of 120 ft-lb

†Values obtained on as-machined specimens

§Furnace treated in open atmosphere for 5 months at 600°F

**Specimens in OMRE bypass line for 13 months at a nominal temperature of 580°F

††Specimens in OMRE dummy element #29 for 8 months at a nominal temperature of 600°F



TABLE B-IV

CORROSION TEST RESULTS ON MATERIALS EXPOSED IN OMRE BYPASS LINE

Specimen Number	Material	Weight Change (mg)	Weight Change/cm ² *	Specimen Number	Material	Weight Change (mg)	Weight Change/cm ² *
A1L	Type 1020 Carbon Steel	-2.3	-0.37	J1L	Type 4130 Alloy Steel	-2.1	-0.34
A2L		-2.3	-0.37	J2L		-2.5	-0.40
A3L		-2.3	-0.37	J3L		-2.6	-0.42
A4L		-1.9	-0.31	J4L		-2.4	-0.39
A5L		-1.4	-0.23	J5L		-2.6	-0.42
A6L		-1.9	-0.31	J6L		-2.5	-0.40
A8L		-1.7	-0.27	J7L		-2.5	-0.40
				J8L		-2.3	-0.38
B1L	Type 304 Stainless Steel	+0.3	+0.05	D1L	Type 1100-0 Aluminum	-1.9	-0.31
B2L		+0.1	+0.02	D2L		-2.6	-0.42
B3L		+0.1	+0.02	D3L		-2.9	-0.47
B4L		-0.1	-0.02	D4L		-4.3	-0.69
B5L		-0.2	-0.03	D5L		-2.2	-0.36
B6L		+0.2	+0.03	D6L		-3.8	-0.61
B8L		-0.1	-0.02	D7L		-1.1	-0.18
C1L	Type 304 Stainless Steel (sensitized for 2 hr at 600°F)	0.0	0.00	E2L	Magnesium	-58.2	-9.38
C2L		-0.2	-0.03	E3L		-58.4	-9.42
C3L		0.0	0.00	E4L		-59.1	-9.53
C4L		-0.2	-0.03	E5L		-59.9	-9.66
C5L		-0.4	-0.06	E6L		-60.0	-9.68
C6L		0.0	0.00	E7L		-60.1	-9.70
C7L		-0.2	-0.03	E2N		-58.4	-9.42
C8L		-0.4	-0.06	F1L	Magnesium Anodized (light)	+9.1	+1.47
H1L	Type 410 Stainless Steel	-0.5	-0.08	F2L		+5.1	+0.83
H2L		-0.5	-0.08	F3L		+5.9	+0.95
H3L		-0.5	-0.08	G1L	Magnesium Anodized (heavy)	-20.0	-3.23
H4L		-0.4	-0.06	G2L		-21.1	-3.41
H5L		0.0	0.00	G3L		-17.7	-2.86
H6L		-0.3	-0.05	A7L/B7L-1	1020 Steel - 304 SS (bimetal)	-1.2	-0.19
H7L		-0.4	-0.06	A7L/B7L-2		-1.3	-0.21
H1N		-1.2	-0.19				

*Surface Area = 6.20 cm²



REFERENCES

1. H. E. Kline, N. J. Gioseffi, and W. Bley, "Dynamic Corrosion in Polyphenyls Under Irradiation," NAA-SR-2046 (May 15, 1958)
2. K. R. Barker and J. W. Mausteller, "High Velocity Corrosion Study in Organic Media," MSA-TR-54 (February 27, 1957)
3. G. C. Milak, "Corrosion and Other Aspects Relative to the Use of Diphenyl As a Coolant-Moderator," ANL-5587 (November 1956)
4. W. K. Boyd et al., "Corrosion Studies in Organic Heat Exchange Media," BMI-1160 (January 28, 1957)
5. C. A. Trilling, "Organic Moderated Reactor Experiment First Progress Report, October, 1955 - July, 1956," NAA-SR-1700 (March 15, 1957)
6. J. C. Wilson and D. S. Billington, Metals 8, No. 5 (1956), 665