Distribution Category: UC-504
Materials

ANL-98/3

ARGONNE NATIONAL LABORATORY
P.O. Box 2528
Idaho Falls, Idaho 83403

THE EBR-II MATERIALS-SURVEILLANCE PROGRAM: IV. Results of SURV-4 and SURV-6

by

W. E. Ruther,* G. O. Hayner,** B. G. Carlson,***
E. R. Ebersole,† and T. R. Allen

Engineering Division
Argonne National Laboratory

January 1998

Part I is ANL-7624
Part II is ANL-7682
Part III is ANL-7937

*Energy Technology Division
**Retired, Hot Fuel Examination Facility, Engineering Division
***Retired, Fuels and Operations Division
†Retired, Analytical Laboratory, Engineering Division

DISCLAIMER

TABLE OF CONTENTS

	Page
ABSTRACT	vii
I. INTRODUCTION	1
II. DOSIMETRY AND EXPOSURE	1
III. RESULTS OF POSTIRRADIATION EXAMINATIONS	5
A. Weight Change	5
B. Density Change	7
C. Metallography	8
D. Hardness	19
E. Strength and Ductility	20
F. Impact Strength	25
G. Bend Tests of Welded Type 304 Stainless Steel	27
H. Measurement of Springs	27
I. Examination of Graphite and Cans	28
1. Can Dimensions	28
2. Graphite Density	29
3. Gas Release From Cans	29
4. Can Material	29
IV. DISCUSSION	30
ACKNOWLEDGMENTS	31
REFERENCES	31

LIST OF FIGURES

		Page
1.	^{54}Mn Activity of Iron Wire that Monitored SURV-4 Flux	2
2.	^{60}Co Activity of Copper Wire that Monitored SURV-4 Flux	3
3.	^{46}Sc Activity of Titanium Wire that Monitored SURV-4 Flux	3
4.	Aluminum Bronze, SURV-4	9
5.	Aluminum Bronze, SURV-6	9
6.	Stellite 6B, SURV-6	10
7.	Inconel X-750, SURV-4	10
8.	Inconel X-750, SURV-6	11
9.	Type 420 Stainless Steel, SURV-4	11
10.	Type 420 Stainless Steel, SURV-6	12
11.	Type T-1 Tool Steel, SURV-4	12
12.	Type T-1 Tool Steel, SURV-6	13
13.	Type 347, SURV-4	13
14.	Type 347, SURV-6	14
15.	Type 416 Stainless Steel, SURV-4	14
16.	Type 416 Stainless Steel, SURV-6	15
17.	Berylco-25, SURV-4	15
18.	Berylco-25, SURV-6	16
19.	Type 304B, SURV-4	16
20.	Type 304B, SURV-6	17
21.	Type 17-4 PH, SURV-4	17
22.	Type 17-4 PH, SURV-6	18
23.	Type 304, SURV-4	18
24.	Type 304, SURV-6	19
25.	Izod Impact Strength of Inconel X750	26
26.	Izod Impact Strength of 17-4 PH Stainless Steel	26

LIST OF TABLES

		Page
I.	Neutron Activation Rates from Flux Wires	4
II.	SURV-4 Maximum Fluences	4
III.	Summary of SURV Exposures	5
IV.	Weight Changes of Hardness Samples from SURV-4 and SURV-6	6
V.	Summary of Density Changes for SURV-4 and SURV-6	7
VI.	Comparative Hardness of Materials	19
VII.	Results of Tensile Tests, SURV-4	21
VIII.	Results of Tensile Tests, SURV-6	23
IX.	Results of Tensile Tests-Control Specimens	24
X.	Results of Bend Tests	27
XI.	Effect of Various Exposures on the Properties of Inconel X750 Springs	28
XII.	Effect of EBR-II Exposure on Graphite Density	30
XIII.	Gas Content of Graphite Cans	30

THE EBR-II MATERIALS-SURVEILLANCE PROGRAM:
IV. Results of SURV-4 and SURV-6

by

W. E. Ruther, G. O. Hayner, B. G. Carlson,
E. R. Ebersole, and T. R. Allen

ABSTRACT

In March of 1965, a set of surveillance (SURV) samples was placed in the EBR-II reactor to determine the effect of irradiation, thermal aging, and sodium corrosion on reactor materials. Eight subassemblies were placed into row 12 positions of EBR-II to determine the effect of irradiation at 370°C. Two subassemblies were placed into the primary sodium basket to determine the effect of thermal aging at 370°C. For both the irradiated and thermally aged samples, one half of all samples were exposed to primary system sodium while one half were sealed in capsules with a helium atmosphere. Fifteen different structural materials were tested in the SURV program. In addition to the fifteen types of metal samples, graphite blocks were irradiated in the SURV subassemblies to determine the effect of irradiation on the graphite neutron shield. In this report, the properties of these materials irradiated at 370°C to a total fluence of 2.2 x 10^{22} n/cm^2 (over 2994 days) are compared with those of similar specimens thermally aged at 370°C for 2994 days in the storage basket of the reactor. The properties analyzed were weight, density, microstructure, hardness, tensile and yield strength, impact strength, and creep.

The Berylco-25 and tantalum specimens exposed to the sodium coolant were the only materials with any significant weight loss. Tantalum also experienced the greatest loss in density as a result of neutron flux. The 300 series stainless steels and tool steel T-1 decreased in density to a lesser degree. Only Berylco-25 showed any significant interaction at the alloy-sodium interface. In general, irradiation caused very little microstructural change in any of the alloys.

The 300 series stainless steels had the most significant hardness increase due to irradiation. The 17-4 precipitation hardened (PH) specimens exhibited the greatest hardness increase due to thermal aging. The effect of fluence on tensile strength varied widely among the different materials, ranging from a loss of 10 percent in ultimate strength for aluminum-bronze and Type 420 stainless steel to a gain of over 100 percent for tantalum. Inconel X-750 and 17-4 PH stainless steel showed substantial loss of low temperature impact strength as a result of irradiation. The notch ductility of EBR-II cover plate material (Type 304 stainless steel) was preserved as evidenced by no fractures at the lowest impact-test temperature. There was no clear pattern for the small changes observed in spring constants of Inconel X-750 springs stored in helium or air, irradiated or thermally aged. However, there was a pronounced increase in spring constant for those springs exposed to reactor sodium.

The density of graphite samples did not change significantly and the cans enclosing the graphite showed no bulging or bowing. Gas pressures in the cans remained below atmospheric.

I. INTRODUCTION

In March of 1965, a set of surveillance (SURV) samples was placed in the EBR-II reactor to determine the effects of irradiation, thermal aging, and sodium corrosion on reactor materials. Eight subassemblies (SURV 1-5 and SURV 8-10), containing 15 alloys used in the primary system of EBR-II, were placed in the EBR-II blanket (Row 12) at 370°C. In addition to the metal samples, these subassemblies also contained a section with shield graphite canned in Type 304 stainless steel. Two other subassemblies (SURV 6-7) were placed in the primary sodium tank storage basket, also at 370°C, to separate thermal and radiation effects. Half of the alloy specimens were exposed directly to reactor sodium, while the remainder were in sealed tubes with a helium atmosphere. The graphite was contained in a 304 stainless steel can with a helium atmosphere. The alloys included Ampco Grade 18 aluminum bronze; Stellite 6B; Inconel X-750; T-1 tool steel; Berylco-25 (beryllium-copper); Types 304, 347, 416, and 420 stainless steel; Type 17-4 PH stainless steel; and tantalum.

The program and experimental methods have been described [1] and the results of SURV-1, -2 and -3 have been reported [1-3]. This report presents the results of examination of SURV-4 (exposed in the reactor) and that of SURV-6 (thermally aged in the sodium tank). In all important aspects, the loading diagrams for these subassemblies are the same as reported [1] for SURV-1.

Although this work was accomplished over 20 years ago, the results were never published. This report is being published now to ensure that the valuable data from the SURV experiments is widely disseminated and to fully document the SURV work prior to analysis of the samples from the last four SURV subassemblies (SURV 7-10).

II. DOSIMETRY AND EXPOSURE

SURV-4 was removed from the reactor grid on May 14, 1973. The SURV-4 subassembly was in reactor position 12C-7 for 2994 days at approximately 370°C. During this time, the reactor logged 64,439 MW days of operation.

SURV-6 was exposed in the primary sodium tank storage basket for 2994 days. Its temperature history was nearly identical to that of SURV-4, but because of the distance from the reactor, it received essentially no neutron irradiation during its exposure.

Three wire flux monitors in the SURV-4 subassembly were used to determine the relative fast flux as a function of axial position in the subassembly. The upper ends of the wires were 10.58 in. above the core centerline. The neutron reaction for each of the wires is:

Iron: $^{54}Fe\ (n,p)\ ^{54}Mn$
Titanium: $^{46}Ti\ (n,p)\ ^{46}Sc$
Copper: $^{63}Cu\ (n,\alpha)\ ^{60}Co$

A 1/4-in. section was cut at 1-in. intervals from each wire, weighed and analyzed by gamma spectrometry on September 27, 1974. The relative reaction rates are plotted in Figs. 1 through 3 and are useful in assessing the relative exposure of SURV-4 samples to fast neutrons as a function of position in the subassembly.

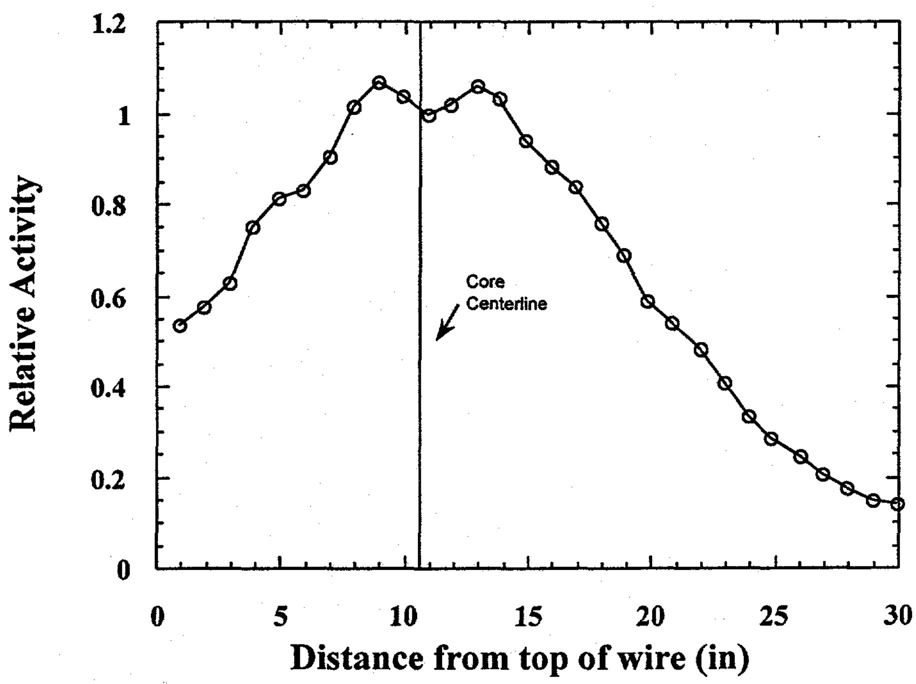


Figure 1. ^{54}Mn Activity of Iron Wire that Monitored SURV-4 Flux

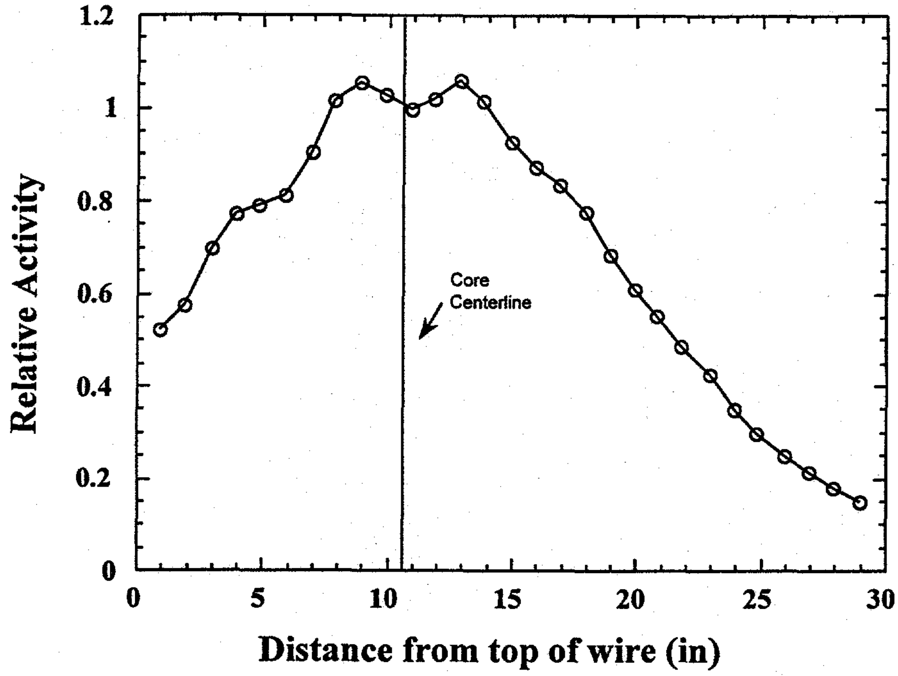


Figure 2. ^{60}Co Activity of Copper Wire that Monitored SURV-4 Flux

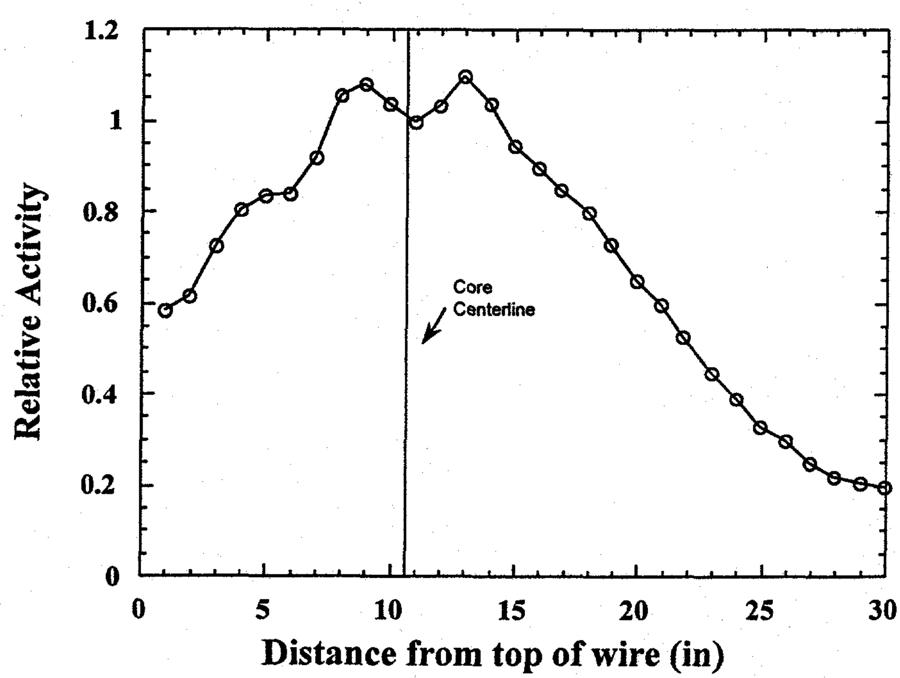


Figure 3. ^{46}Sc Activity of Titanium Wire that Monitored SURV-4 Flux

TABLE I. Neutron Activation Rates from Flux Wires

Reaction	Activity, d/s/g of Wire 9/27/74	62.5 MWt Equilibrium Disintegration Rate, d/s/g Target Atom	62.5 MWt Equilibrium Activation Rate, atoms/sec per Target Atom
^{54}Fe (n,p) ^{54}Mn	2.86×10^7	6.20×10^8	3.05×10^{-13}
^{46}Ti (n,p) ^{46}Sc	3.23×10^5	3.41×10^9	4.53×10^{-14}
^{63}Cu (n,α) ^{60}Co	2.06×10^6	1.47×10^7	1.53×10^{-15}

TABLE II. SURV-4 Maximum Fluences

Energy Level, MeV	Fluence, n/cm^2
>3.38	0.37×10^{20}
>1.35	2.30×10^{20}
>0.82	7.20×10^{20}
>0.11	1.20×10^{22}
Total	2.20×10^{22}

The equilibrium activation rate, atoms/second per target atom, at 62.5 MWt reactor power was calculated from the wire activity data at the core centerline where the relative activity is shown as unity. To account for the intermittent operation of the reactor, the quantity $(1-e^{-\lambda t_1})(e^{-\lambda t_2})$ was evaluated for each reactor run, where t_1 = irradiation time, and t_2 = decay time from end of irradiation to counting time. The values of this quantity were summed to obtain the fraction of saturation at counting time. The activation rates at 62.5 MWt are shown in Table I.

The approximate neutron fluences for SURV-4 as calculated by diffusion theory are given in Table II.

A comparison of SURV-4 with the previous SURV subassemblies is shown in Table III. Four rows of uranium blanket subassemblies had been replaced with stainless steel since SURV-3 was removed. The stainless steel reflector provided a somewhat more "leaky" core, and, hence, a slight increase in fluence for the same amount of MWd.

TABLE III. Summary of SURV Exposures

SURV	MWd	Total Fluence, n/cm^2
1	11541	9.0×10^{20}
2	26274	3.0×10^{21}
3	41111	1.2×10^{22}
4	64439	2.2×10^{22}

III. RESULTS OF POSTIRRADIATION EXAMINATIONS

A. Weight Change

To determine the corrosion resistance of core structural materials in the primary system sodium, weight loss was measured. Weight loss was measured in samples exposed to primary system sodium and in samples exposed to helium. The weight-change data (obtained using hardness sample cylinders) are summarized in Table IV. Each exposed sample was ultrasonically cleaned prior to weighing.

Similar to the results from the earlier SURV subassemblies, only the Berylco-25 and tantalum specimens exposed to the sodium coolant showed any significant weight losses. The greater losses for Berylco in SURV-4 compared with SURV-6 are probably due to the greater rate of circulation of sodium through the subassembly in the reactor position as compared with the one in the basket. Because of the serious corrosion indicated by the weight loss, Berylco-25 and unclad tantalum components are no longer used in the reactor, although there are still specimens exposed to the coolant in the remaining SURV subassemblies.

One specimen each of Ampco-18, tool steel T-1 and Type 420 stainless steel showed a larger weight loss than other samples of the same material and irradiation conditions. Similar atypical results were noted for one specimen each of Ampco-18 and tool steel in SURV-2, and for one specimen each of tool steel, Type 17-4 PH and Type 304 stainless steel in SURV-3. These atypical results are likely the result of experimental error.

TABLE IV. Weight Changes of Hardness Samples from SURV-4 and SURV-6

Material	Weight Change,[a] mg, SURV-4		Weight Change,[a] mg, SURV-6	
	Sodium-exposed Samples	Helium-exposed Samples	Sodium-exposed Samples	Helium-exposed Samples
Aluminum bronze—Ampco Grade 18	(A1) –0.1 (A2) –22.6[c] (A3) +1.0 (A4) –0.7	(A5) +0.3 (A6) +0.8 (A7) +1.9 (A8) +0.4	(A1) –0.6 (A2) –0.7 (A3) –0.6 (A4) –1.0	(A5) +0.2 (A6) +0.4 (A7) +0.6 (A8) +0.5
Stellite 6B	(B1) +0.1 (B2) –1.8 (B3) –0.2 (B4) +0.1	(B5) +0.1 (B6) +0.2 (B7) +0.3 (B8) +0.2	(B1) –0.1 (B2) –0.3 (B3) +0.1 (B4) –0.1	(B5) –0.1 (B6) 0.0 (B7) –0.1 (B8) +0.2
Inconel X-750 (Heat-treated to hardness of Rockwell C 55-60)	(C1) +0.6 (C2) +1.1 (C3) +0.8 (C4) +0.6	(C5) +0.1 (C6) +0.1 (C7) +0.1 (C8) +0.2	(C1) +0.3 (C2) +0.1 (C3) +0.1 (C4) 0.0	(C5) 0.0 (C6) +0.2 (C7) +0.1 (C8) +0.2
Type 420 stainless steel (Heat-treated to hardness of Rockwell C 40-45)	(D1) –6.2[c] (D2) +0.1 (D3) +0.1 (D4) +0.2	(D5) +0.1 (D6) +0.1 (D7) +0.1 (D8) +0.1	(D1)[d] (D2) –0.1 (D3) +0.1 (D4) –0.1	(D5) 0.0 (D6) +0.2 (D7) +0.1 (D8) +0.2
Tool steel T-1 (Heat-treated to hardness of Rockwell C 55-60)	(E1) –2.9 (E2) –2.3 (E3) –0.1 (E4) +0.1	(E5) +0.3 (E6) +0.3 (E7) +0.2 (E8) +0.3	(E1) –20.8[c] (E2) –0.9 (E3) –0.1 (E4) +0.1	(E5) +0.5 (E6) +0.2 (E7) –0.4 (E8) +0.1
Type 347 stainless steel	(F1) –0.3 (F2) 0.0 (F3) +0.1 (F4) +0.1	(F5) +0.1 (F6) 0.0 (F7) 0.0 (F8) +0.1	(F1) –0.7 (F2) –1.2 (F3) 0.0 (F4) –0.1	(F5) +0.1 (F6) 0.0 (F7) +0.1 (F8) +0.2
Type 416 stainless steel (Heat-treated to hardness of Rockwell C 30-34)	(G1) +4.8 (G2) +0.4 (G3) +0.2 (G4) +0.4	(G5) +0.2 (G6) +0.1 (G7) +0.2 (G8) +0.2	(G1) –0.2 (G2) +0.3 (G3) +0.3 (G4) +0.2	(G5) +0.2 (G6) +0.2 (G7) +0.1 (G8) +0.2
Beryllium Copper—Berylco-25 (Heat-treated to hardness of Rockwell C 41-45)	(H1) –882.4 (H2) –336.4 (H3) –759.8 (H4) –538.7	(H5) +0.2 (H6) +0.2 (H7) +0.1 (H8) +0.1	(H1) –10.3 (H2) –0.6 (H3) –10.3 (H4) –1.2	(H5) +0.1 (H6) 0.0 (H7) 0.0 (H8) +2.5[c]
Type 304 stainless steel with boron	(I1) –0.5 (I2) –0.2 (I3) +0.1 (I4) –0.4	(I5) +0.1 (I6) +1.2 (I7) +0.3 (I8) 0.0	(I1) –0.3 (I2) –0.2 (I3) –0.3 (I4) +0.2	(I5) –0.1 (I6) 0.0 (I7) 0.0 (I8) +0.3
Type 17-4 PH stainless steel (Heat-treated to hardness of Rockwell C 36-41)	(J1) –0.2 (J2) +0.1 (J3) –0.1 (J4)[d]	(J5) +0.5 (J6) +0.2 (J7) +0.2 (J8) +0.1	(J1) +5.1[c] (J2) –0.2 (J3) –0.2 (J4) –0.1	(J5) 0.0 (J6) 0.0 (J7) –0.1 (J8) +0.3

TABLE IV. Weight Changes of Hardness Samples from SURV-4 and SURV-6 (Continued)

Material	Weight Change,[a] mg, SURV-4		Weight Change,[a] mg, SURV-6	
	Sodium-exposed Samples	Helium-exposed Samples	Sodium-exposed Samples	Helium-exposed Samples
Type 304 stainless steel	(K1) 0.0 (K2) –1.4[c] (K3) +0.2 (K4) +0.4	(K5) +0.1 (K6) +0.1 (K7) +0.2 (K8) –4.3[c]	(K1) +0.1 (K2) +0.1 (K3) +0.3 (K4) +0.3	(K5) 0.0 (K6) +0.1 (K7) +1.3 (K8) 0.0
Tantalum	(M1) –16.1 (M2) –21.3 (M3) –37.8 (M4) –20.6	(M5) +2.6 (M6) +6.7 (M7) +2.8 (M8) +2.1	(M1) –14.3 (M2) –16.1 (M3) –12.9 (M4) –26.6	(M5) +0.2 (M6) +0.8 (M7) +0.2 (M8) +0.4

[a]Cylindrical exposed area was 7.8 cm^2; total area 9.7 cm^2.

[b]Sample numbers are shown in parentheses.

[c]Sample damage in handling and/or weighing error is strongly suspected.

[d]Not weighed.

B. Density Change

Density was measured to determine the propensity of core materials to swell. Density determinations were performed on those hardness-corrosion specimens which had received the largest fluence in SURV-4 and on those from equivalent locations in SURV-6. Except in the case of tantalum specimens, the same platinum-iridium standard used in establishing the pre-exposure densities was available for checking these measurements. The results are summarized in Table V. The effect of irradiation or thermal aging on density was small for all alloys. The percentage decrease in density due to irradiation was largest for tantalum, T-1 tool steel, 304 stainless steel, and 304 plus boron, although each changed by less than one-half percent. Many of the other alloys showed much smaller changes.

TABLE V. Summary of Density Changes for SURV-4 and SURV-6

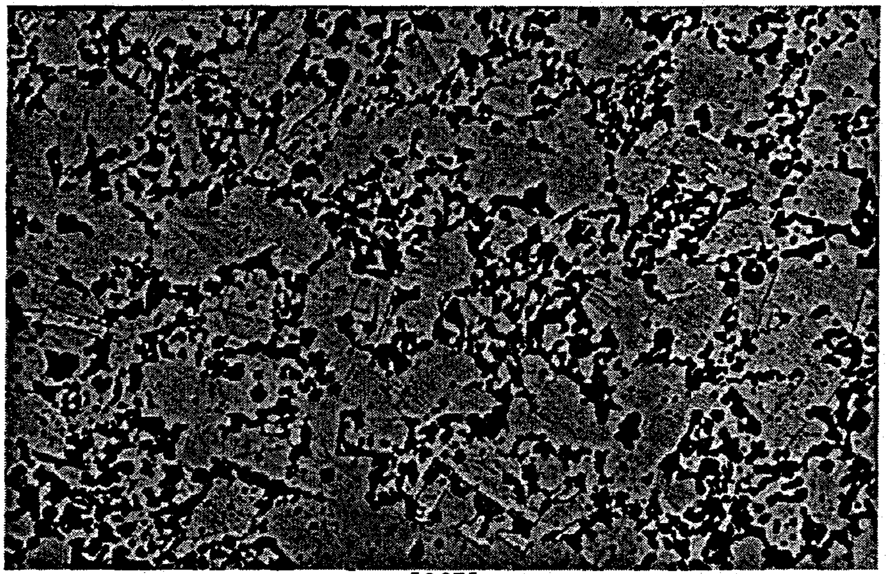
Material	Density Change			
	SURV-4 (Reactor)		SURV-6 (Basket)	
	g/cm^3	%	g/cm^3	%
Ampco 18	-0.041	-0.55	-0.039	-0.52
Stellite 6B	-0.006	-0.07	-0.010	-0.12
Inconel X-750	-0.009	-0.11	-0.010	-0.12
Type 420 stainless steel	-0.009	-0.12	-0.009	-0.12

TABLE V. Summary of Density Changes for SURV-4 and SURV-6
(Continued)

Material	Density Change			
	SURV-4 (Reactor)		SURV-6 (Basket)	
	g/cm^3	%	g/cm^3	%
Tool Steel T-1	-0.032	-0.37	-0.016	-0.19
Type 347 stainless steel	-0.024	-0.30	-0.014	-0.18
Type 416 stainless steel	-0.010	-0.13	-0.011	-0.14
Berylco-25	-0.010	-0.12	+0.006	+0.07
Borated Type 304 stainless steel	-0.030	-0.39	-0.013	-0.17
Type 17-4 PH stainless steel	-0.014	-0.18	-0.019	-0.25
Type 304 stainless steel	-0.029	-0.37	-0.010	-0.13
Tantalum	-0.055	-0.33	+0.035	+0.21

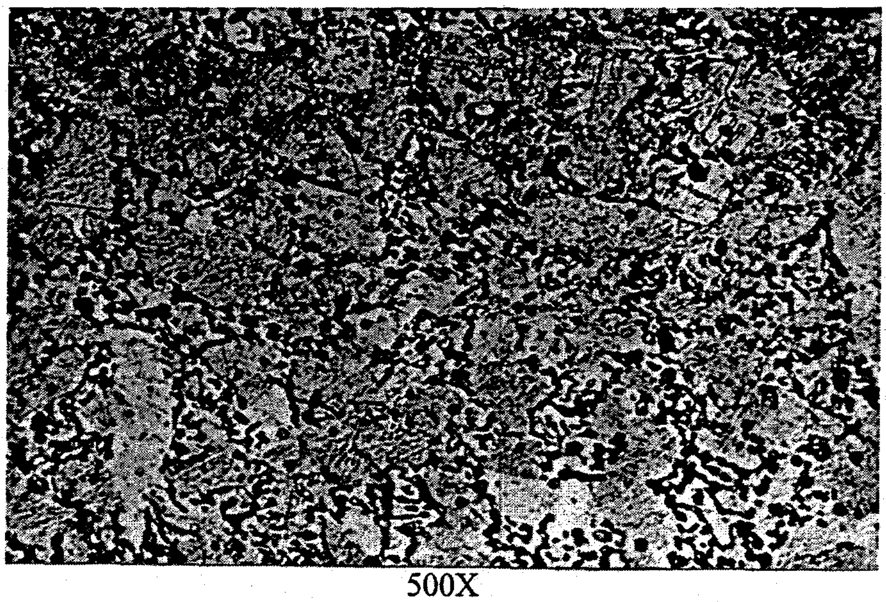
C. Metallography

To determine how irradiation, thermal aging, and corrosion affect core structural materials, the microstructure of the samples was examined. The metallographic microstructure of specimens from SURV-4 and SURV-6 are shown in Figs. 4 through 24. Irradiated specimens of Stellite 6B and tantalum could not be examined due to their intense radioactivity. Only Berylco-25 showed any significant interaction at the alloy-sodium interface. In general, there was very little change in microstructure on the scale visible in optical microscopy attributable to irradiation. The changes in hardness and tensile properties indicate smaller scale microstructural changes did occur, but no transmission electron microscopy (TEM) was performed. An anomalous result was obtained with the Type 347 stainless steel specimen exposed in SURV-4. A partially sensitized structure was obtained which cannot be explained from its known history. It is believed that an incorrectly heat-treated specimen was accidentally incorporated in the subassembly.



500X

Figure 4. Aluminum Bronze, SURV-4
($NH_4OH - H_2O_2$ etchant)



500X

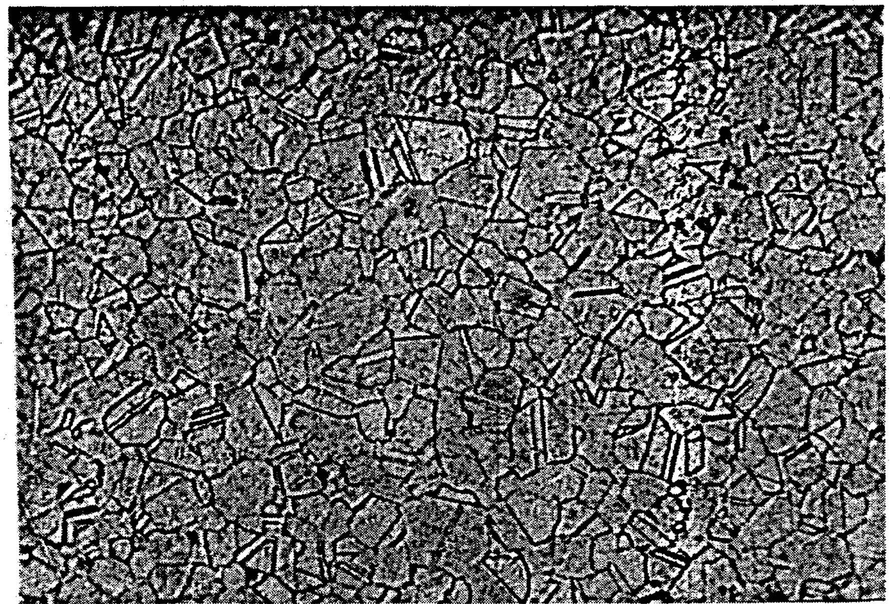
Figure 5. Aluminum Bronze, SURV-6
($NH_4OH - H_2O_2$ etchant)

500X

Figure 6. Stellite 6B, SURV-6
(HNO_3 – HCl – H_2O etchant)

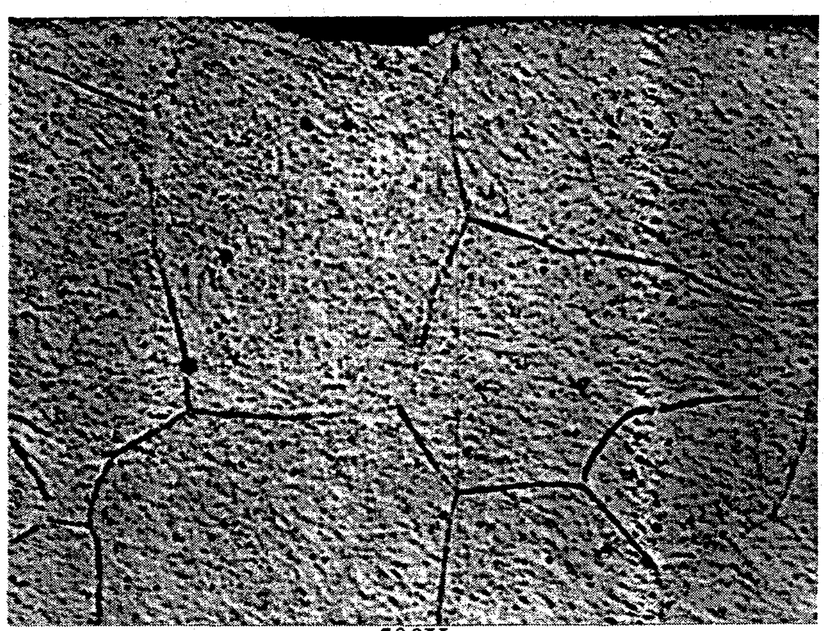
500X

Figure 7. Inconel X-750, SURV-4
(HNO_3 – HCl – H_2O etchant)



500X

Figure 8. Inconel X-750, SURV-6
(HNO_3 – HCl – H_2O etchant)

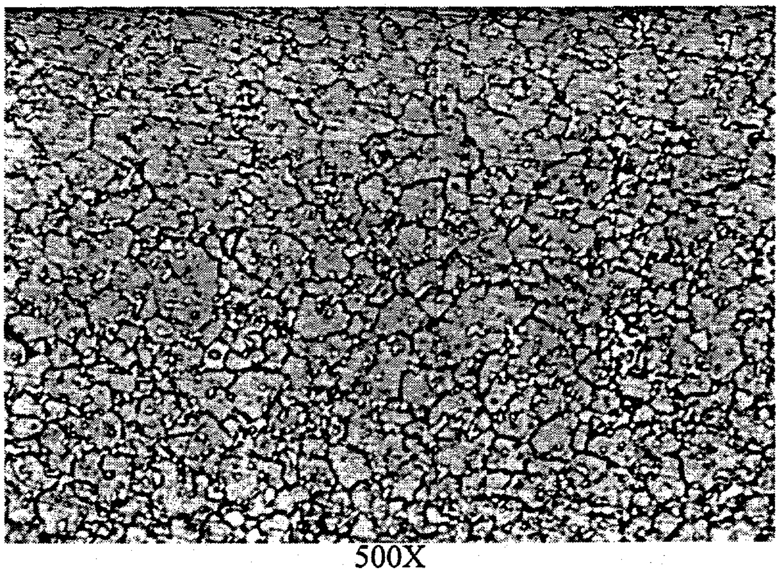


500X

Figure 9. Type 420 Stainless Steel, SURV-4
(HNO_3 – HCl – H_2O etchant)

500X

Figure 10. Type 420 Stainless Steel, SURV-6
(HNO_3 etchant)

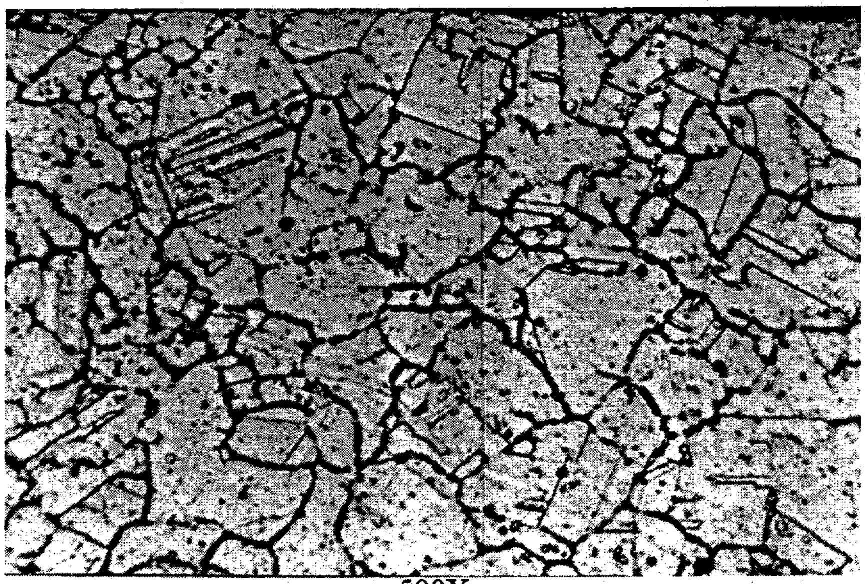


500X

Figure 11. Type T-1 Tool Steel, SURV-4

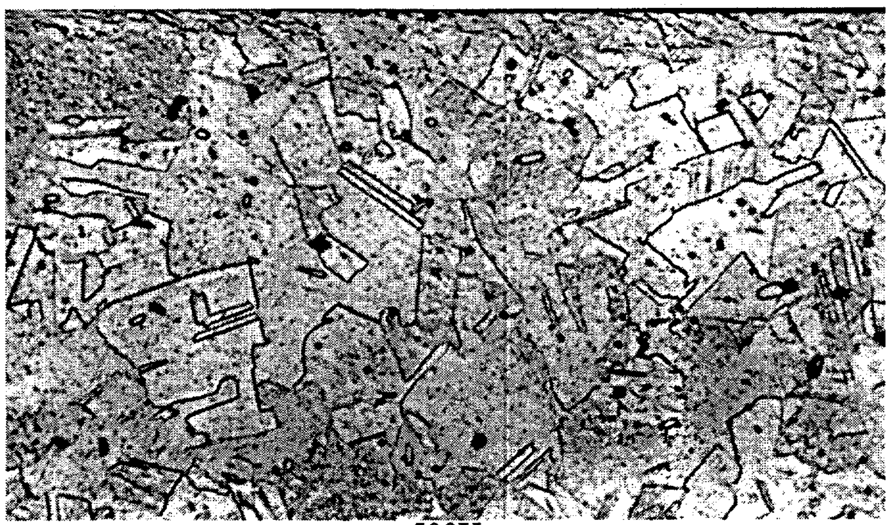
500X

Figure 12. Type T-1 Tool Steel, SURV-6
(HNO_3 etchant)



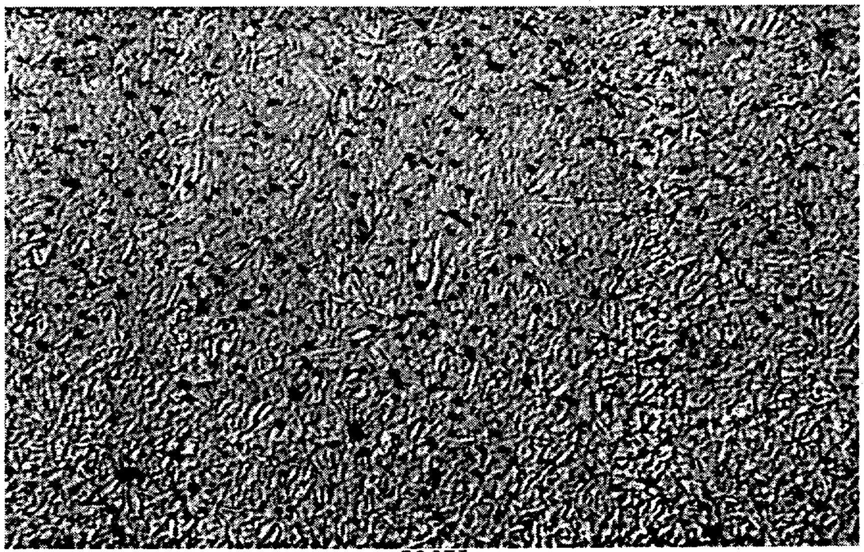
500X

Figure 13. Type 347, SURV-4
(Oxalic etchant)



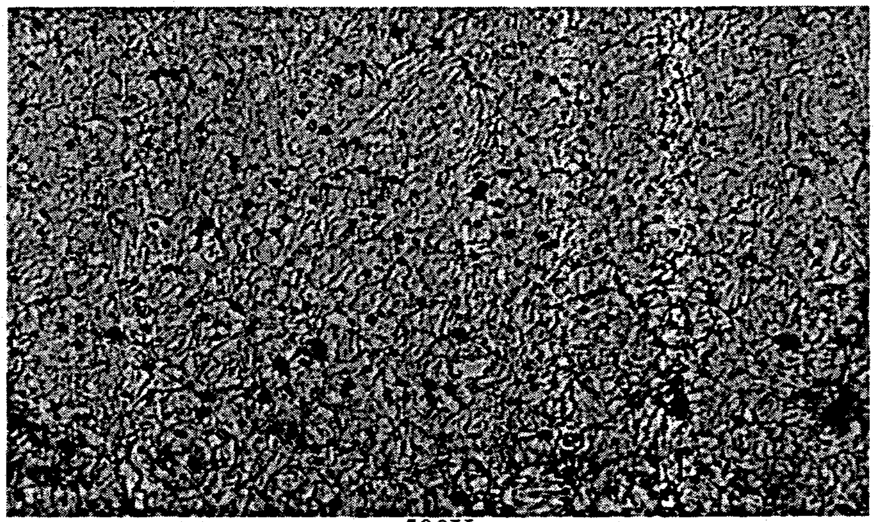
500X

Figure 14. Type 347, SURV-6
(Oxalic etchant)



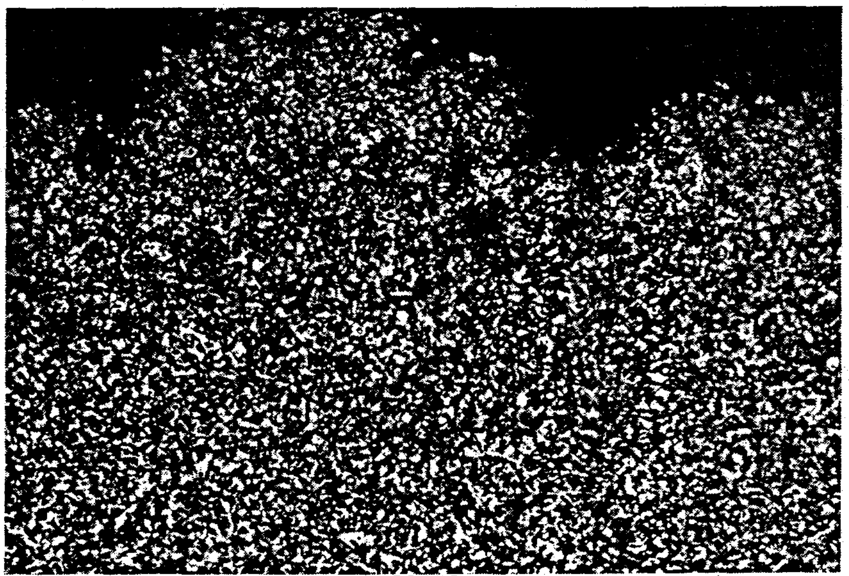
500X

Figure 15. Type 416 Stainless Steel, SURV-4
($FeCl_3$ etchant)



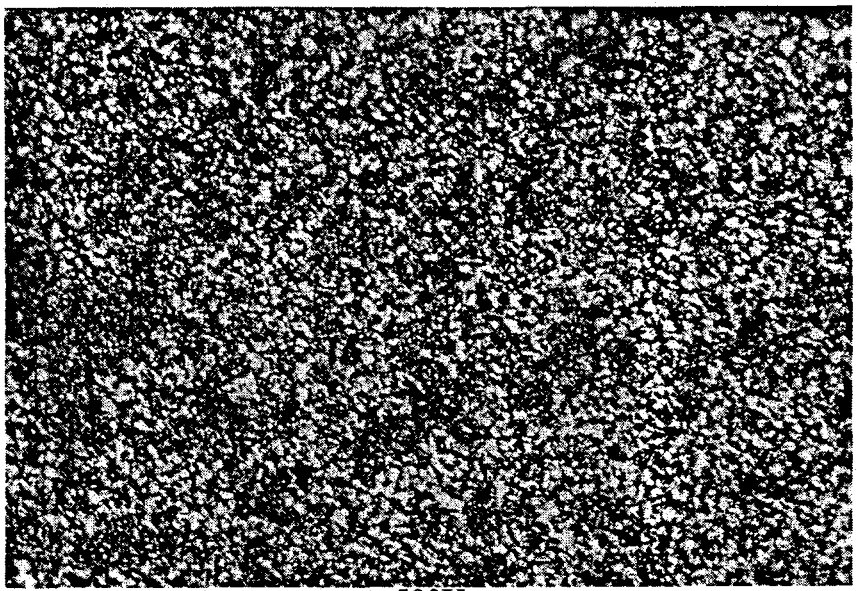
500X

Figure 16. Type 416 Stainless Steel, SURV-6
($FeCl_3$ etchant)



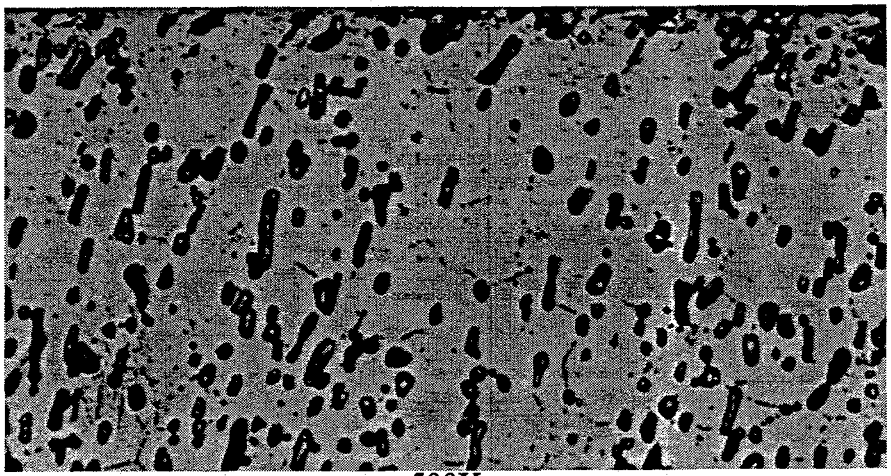
500X

Figure 17. Berylco-25, SURV-4
(NH_4OH – H_2O_2 etchant)



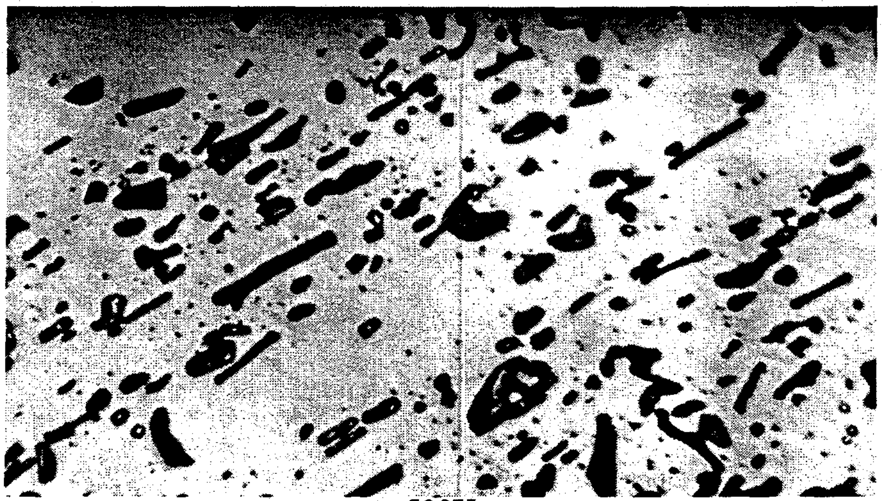
500X

Figure 18. Berylco-25, SURV-6
($NH_4OH - H_2O_2$ etchant)



500X

Figure 19. Type 304B, SURV-4
(Oxalic etchant)

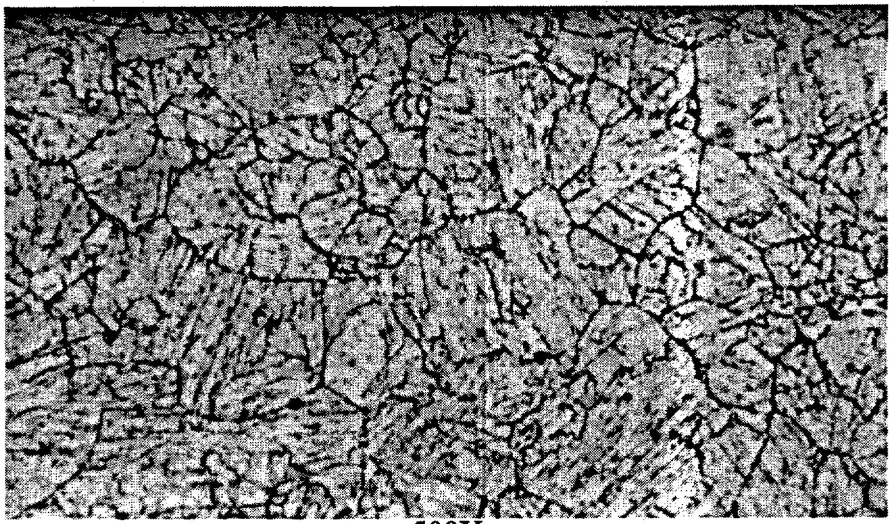


500X

Figure 20. Type 304B, SURV-6
(Oxalic etchant)

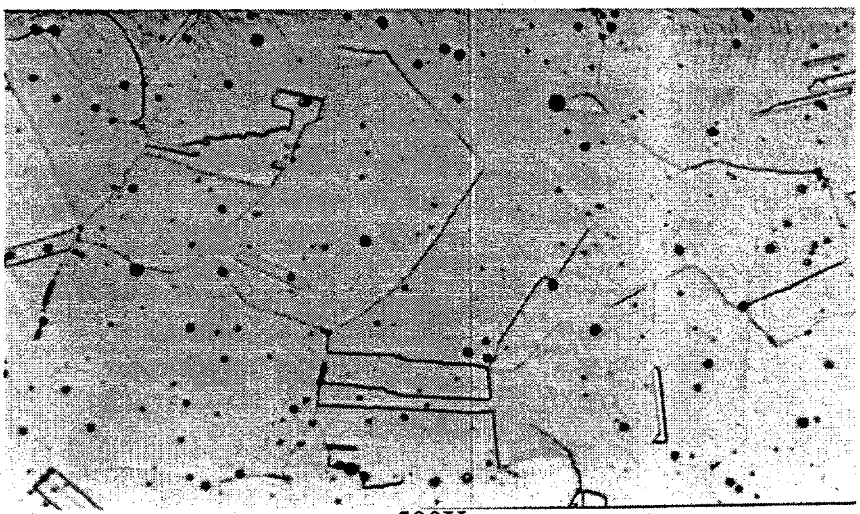
500X

Figure 21. Type 17-4 PH, SURV-4
(HCl etchant)



500X

Figure 22. Type 17-4 PH, SURV-6
(HCl etchant)



500X

Figure 23. Type 304, SURV-4
(Oxalic etchant)

500X

Figure 24. Type 304, SURV-6
(Oxalic etchant)

D. Hardness

Hardness was measured to estimate changes in strength. Table VI presents hardness measurements made on SURV specimens. Microhardness was measured on two samples from each material, one exposed in sodium, the other in helium. Ten indentations were averaged for each reported value. No significant variation in hardness was noted between sodium and helium exposure. Therefore, the environment did not effect the material hardening. Aluminum bronze and Berylco-25 alloys experienced hardness changes due to thermal aging (SURV-6). The austenitic stainless steels showed little change in hardness as a result of the high temperature, but were hardened by the neutron flux. In contrast, thermally aged 17-4 PH specimens exhibited a greater hardness increase than irradiated 17-4 PH.

TABLE VI. Comparative Hardness of Materials

Material	Vickers Hardness (HV)		
	As Received	SURV-4	SURV-6
Aluminum Bronze	180-197	209[b] 207[c]	222[b] 210[c]
Haynes Stellite 6B	360-389	Too radioactive for existing equipment	385 390
Inconel X750	363-375	378 385	362 351

TABLE VI. Comparative Hardness of Materials
(Continued)

Material	Vickers Hardness (HV)		
	As Received	SURV-4	SURV-6
Type 420 SS	365-410	365 370	380 400
Tool Steel T-1	640-700	640 640	685 688
Type 347 SS	150-160	234 230	149 154
Type 416 SS	285-315	320 320	310 310
Berylco 25	357-378	144 138	148 144
Type 304 with Boron	195-220	348 345	227 226
17-4 PH	325-375	415 403	454 460
Type 304 SS	145-158	252 257	154 148

[a]Results obtained by averaging 10 indentations/sample.

[b]First value for each alloy for specimen exposed in sodium.

[c]Second value for each alloy for specimen exposed in helium.

E. Strength and Ductility

Tensile tests were performed to determine changes in strength and ductility. Room temperature and 370°C tensile tests were performed using a cross-head speed of 0.02 in./m. Specimens 1 through 4 had been exposed to the sodium coolant, while 5 through 8 had been enclosed in helium-filled capsules. The experimental results are presented in Tables VII and VIII. The values for the control (no exposure) specimens are given in Table IX.

In the earlier examination of SURV-3, it was determined that T-1 tool steel and 17-4 PH specimens were so embrittled that they broke in their threaded portion within the grips. These alloys were not tested since the broken pieces could not be removed from the tensile grips used.

TABLE VII. Results of Tensile Tests, SURV-4

Material	Specimen No.	Environment	Test Temperature, °C	UTS,* ksi	YS,** ksi	Elongation %	Reduction in Area, %
Aluminum	1	Na	370	42.6	38.6	7[a]	14
Bronze	3		370	44.4	39.7	12[a]	17
	5	He	370	43.9	41.7	6.5[a]	17
	7		370	54.0	50.9	--	14
	2	Na	25	95.0	53.4	15[b]	17
	4		25	95.9	56.8	5[a]	17
	6	He	25	95.5	58.3	12[b]	12
	8		25	91.2	54.2	10	8
Inconel X750	1	Na	370	169	138	11[b]	26
	3		370	162	124	19	28
	5	He	370	166	138	10[a]	41
	7		370	173	133	20	33
	2	Na	25	184	162	10	14
	4		25	173	135	17[b]	28
	6	He	25	180	156	12	34
	8		25	178	143	13	29
Type 420	1	Na	370	151	131	16	46
Stainless	3		370	155	135	--[c]	44
Steel	5	He	370	144	124	19	43
	7		370	192	173	13	43
	2	Na	25	187	168	11	46
	4		25	162	136	8[b]	51
	6	He	25	184	164	12	46
	8		25	167	141	18[b]	46
Type 304	1	Na	370	97.5	84.4	17	59
Wrought	3		370	94.1	84.4	13[b]	54
Stainless	5	He	370	106	98.6	9[b]	52
Steel	7		370	89.4	76.2	21	58
	2	Na	25	127	104	49	75
	4		25	133	111	42	72
	6	He	25	111	83.1	57	75
	8		25	95.0	83.4	22	59

TABLE VII. Results of Tensile Tests, SURV-4
(Continued)

Material	Specimen No.	Environment	Test Temperature, °C	UTS,* ksi	YS,** ksi	Elongation %	Reduction in Area, %
Type 304	1	Na	370	92.5	85.3	3[b]	30
Stainless	3		370	75.1	50.7	11[b]	26
Steel +	5	He	370	89.4	81.7	6[b]	43
Type 308	7		370	74.6	51.2	14[b]	43
Weld	2	Na	25	107	80.9	19[a]	59
	4		25	109	84.0	28[b]	47
	6	He	25	109	89.6	23[a]	59
	8		25	86.3	75.0	7[b]	40
Type 304	1	Na	370	77.3	60.9	24[b]	54
Stainless	3		370	83.1	71.2	26	70
Steel –	5	He	370	77.1	60.4	33	66
EBR-II	7		370	80.5	67.2	29	73
Cover Plate	2	Na	25	101	76.9	52[a]	77
	4		25	94.8	62.4	53[a]	85
	6	He	25	78.2	65.9	28	61
	8		25	73.1	55.5	32[b]	68
Tantalum	1	Na	370	119	119	15	49
	3		370	118	117	18	60
	5	He	370	125	125	--[a]	53
	7		370	105	103	--[a]	45
	2	Na	25	138	138	12	60
	4		25	128	124	18	53
	6	He	25	146	143	20	53
	8		25	140	138	20	65

*Ultimate Tensile Strength

**Yield Strength

[a]Elongation inaccurate, broke outside gauge marks.

[b]Elongation suspect, broke at gauge marks.

[c]No gauge marks identifiable.

TABLE VIII. Results of Tensile Tests, SURV-6

Material	Specimen No.	Environment	Test Temperature, °C	UTS, ksi	YS, ksi	Elongation %	Reduction in Area, %
Aluminum Bronze	1	Na	370	57.0	50.8	16[a]	40
	3		370	66.4	56.1	25[a]	42
	5	He	370	38.9	35.8	13[a]	66
	7		370	50.0	45.4	12[a]	54
	2	Na	25	102	61.6	16	14
	4		25	104	64.4	16[b]	12
	6	He	25	101	62.7	12[b]	23
	8		25	103	65.4	13[b]	13
Inconel X750	1	Na	370	150	91.2	25[b]	23
	3		370	154	95.0	28	32
	5	He	370	159	93.4	25[b]	33
	7		370	160	95.2	36	42
	2	Na	25	170	104	20[a]	32
	4		25	164	102	17[a]	23
	6	He	25	170	106	33	23
	8		25	169	102	29[a]	31
Type 420 Stainless Steel	1	Na	370	172	145	17	44
	3		370	186	161	17	44
	5	He	370	170	145	3	24
	7		370	173	152	3	53
	2	Na	25	210	168	9[b]	44
	4		25	212	179	--[c]	44
	6	He	25	206	179	15[b]	42
	8		25	198	176	14	51
Type 304 Wrought Stainless Steel	3	Na	370	80.8	65.1	28	62
	5	He	370	87.2	73.1	25	64
	7		370	80.5	63.6	14	61
	4	Na	25	122	104	36	67
	6	He	25	115	91.2	51	75
	8		25	126	105	49	75
Type 304 Stainless Steel + Type 308 Weld	3	Na	370	67.7	31.0	16[a]	53
	5	He	370	68.8	30.0	19[a]	47
	7		370	67.6	28.9	19[a]	52
	2	Na	25	91.2	44.0	30[a]	65
	4		25	92.3	44.3	33[a]	67
	6	He	25	90.5	44.3	30[a]	68
	8		25	92.8	47.1	30[a]	66
Type 304 Stainless Steel – EBR-II Cover Plate	1	Na	370	63.4	18.6	56	75
	3		370	62.4	19.8	39[a]	79
	5	He	370	63.2	23.3	52	75
	7		370	63.6	29.0	51	75
	2	Na	25	87.4	38.0	87	86
	4		25	85.8	32.1	80[a]	89
	6	He	25	63.2	21.6	37[a]	73
	8		25	56.9	30.0	43	72

TABLE VIII. Results of Tensile Tests, SURV-6
(Continued)

Material	Specimen No.	Environment	Test Temperature, °C	UTS, ksi	YS, ksi	Elongation %	Reduction in Area, %
Tantalum	1	Na	370	48.1	31.0	46	73
	3		370	45.6	29.0	50	62
	5	He	370	49.1	23.3	--[a]	68
	7		370	62.7	38.8	--[a]	62
	2	Na	25	56.8	46.7	38	71
	4		25	55.0	52.6	52	72
	6	He	25	58.7	52.5	59	73
	8		25	55.1	55.1	46	71

[a]Elongation inaccurate, broke outside gauge marks.
[b]Elongation suspect, broke at gauge marks.
[c]No gauge marks identifiable.

TABLE IX. Results of Tensile Tests – Control Specimens

Material	Test Temperature °F	UTS, ksi	YS, ksi	Elongation %	Reduction in Area, %
Aluminum Bronze	370	49.3	40.4	20[a]	48
	25	103	52.2	25	60
	(25)[b]	(103)	(49)	(30)	(41)
Inconel X750	370	159	92.0	23	9
	25	169	102	17[a]	23
	(25)	(170)	(96)	(24)	(24)
Type 420 Stainless Steel	370	191	170	13[b]	48
	25	(Testing Machine Inadequate to Break Specimen)			
	(25)	(224)	(170)	6	36
Type 304 Stainless Steel (Wrought)	370	91.4	82.0	18[b]	49
	25	124	(105)	47	75
	(25)	(111)	(85)	(40)	58
Type 304 Stainless Steel + Type 308 Weld	370	66.6	33.6	19[a]	56
	25	94.6	43.4	33	59
	(25)	(88)	(48)	(38)	(59)
Tantalum	(25)	(51)	(36)	(57)	(95)

[a]Value inaccurate; specimen broke outside of gauge marks.
[b]Value suspect; specimen broke at gauge marks.

A comparison of SURV-6 specimens and the controls shows no significant and consistent differences, except that the extended time at 370°C raised the yield strengths of tantalum and aluminum bronze.

The effect of fluence (SURV-4 vs. SURV-6) varied widely among the different materials, ranging from a loss of 10 percent in ultimate strength for aluminum bronze and Type 420 stainless steel to a gain of over 100 percent for tantalum. Type 304 stainless steel and Inconel X750 showed intermediate gains of about 20 percent and 7 percent, respectively, in ultimate strength. These results continue the trends set in earlier SURV examinations. The ductility of all materials tested decreased and showed no marked difference between samples exposed in sodium and in helium.

F. Impact Strength

Changes in ductility were determined using impact tests. Impact tests were performed (by Aerojet Nuclear), using a Warner-Swazey Model BLI impact tester designed for subsize Izod samples. It had a maximum impact-energy capacity of 16 ft-lb delivered at 11.4 ft/s. Each sample was heated or cooled to the desired temperatures, placed in the machine, and subjected to three tests (each Izod sample has three notches [1]).

Data for Inconel X-750 and 17-4 PH stainless steel are shown in Figs. 25 and 26, respectively. Although fluence for these materials in SURV-4 ranged from 1.4×10^{20} to 2.6×10^{20} n/cm^2 (>0.82 MeV), scatter of the data masked any effect due to this variation. Also, there was no discernible difference between specimens exposed in sodium or helium, so data for all samples are plotted without differentiation.

The impact strength of Inconel X750 is significantly increased by thermal aging, but the combined effect of temperature and irradiation is to decrease impact strength. However, both thermal aging and irradiation independently reduced the impact strength of 17-4 PH.

The notch ductility of the EBR-II cover-plate material (Type 304 stainless steel) was preserved. No fractures occurred at the lowest test temperature (-100°F). Tests at higher temperatures were not run for this material.

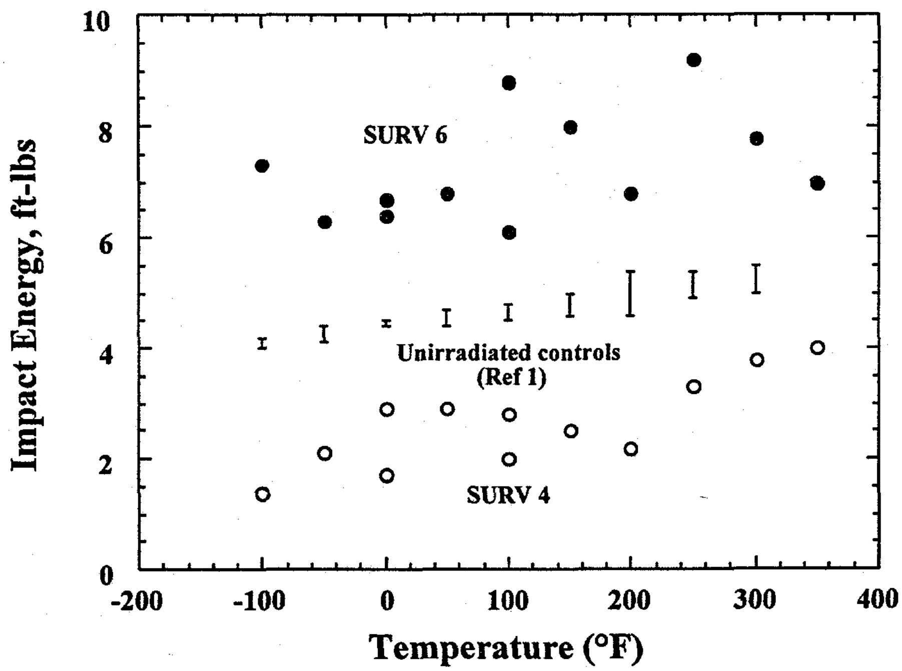


Figure 25. Izod Impact Strength of Inconel X750

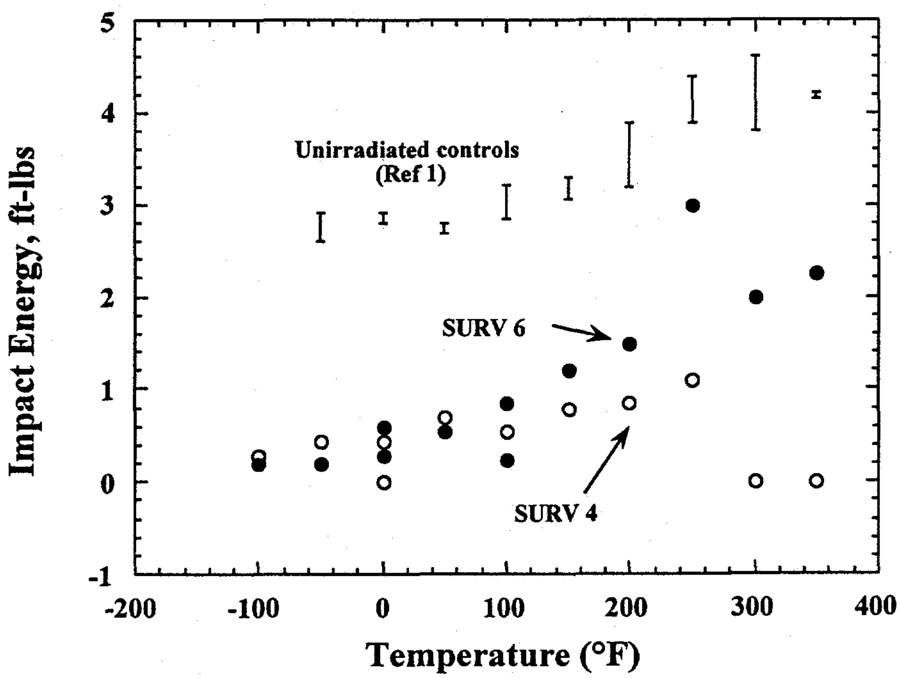


Figure 26. Izod Impact Strength of 17-4 PH Stainless Steel

G. Bend Tests of Welded Type 304 Stainless Steel

To determine the strengthening effect of fluence on 304 stainless steel, bend test samples were tested at ambient temperature supported on round pins on 2-in. centers, with the load applied perpendicularly to the center of the 3.75 x 0.424-in. face by a third round pin. Crosshead speed was 0.2 in./min. No sample fractured during the test. The numerical results, shown in Table X, illustrate the effect of fluence. The values for SURV-6 were essentially the same as those for unexposed specimens [1].. Visual examination of the bent specimens showed no large cracks or other abnormalities.

H. Measurement of Springs

To investigate creep and stress relaxation, irradiated and thermally aged springs were examined. Restrained springs of Inconel X750 were exposed in EBR-II (one set in sodium, one in helium). A set of control springs were stored at room temperature. The sets were prepared originally with calculated preloads of approximately 2.5, 5 and 7.5 pounds. Spring constants were determined for each spring. Similar measurements were made recently on the exposed sets and the controls. The results are shown in Table XI.

TABLE X. Results of Bend Tests

SURV	Specimen No. Type 304 SS (Welded)	Maximum Force*-lb
4	1	378
	2	347
	3	365
	4	361
6	1	273
	2	277
	3	256
	4	275

*Maximum load occurred at approximately 0.4 in. of crosshead travel.

TABLE XI. Effect of Various Exposures on the Properties of Inconel X750 Springs

Location	Calculated Preload, lb	Change in Preload, %*		Change in Spring Constant, %*	
		He	Na	He	Na
SURV-4 (~370°C plus fluence)	2.5	-48	-45	+11	+11
	5	-48	-39	+4	+14
	7.5	-49	-44	+1	+40
SURV-6 (370°C, no fluence)	2.5	-40	-43	+1	+5
	5	-22	-12	0	+19
	7.5	-14	-15	-2	+60
CONTROL SET (Room Temperature)		(Air)		(Air)	
	2.5	-37		0	
	5	-27		+5	
	7.5	-19		+10	

*Each value is the average of three springs.

Comparing preloads, exposure in the reactor basket was roughly equivalent to storage in air. Exposure to neutron flux increased the loss of preload, but there was no significant difference between sodium and helium environments.

No clear pattern emerged for the small changes in spring constants of those springs stored in helium or air, fluence or no fluence. However, unirradiated springs exposed to reactor sodium, particularly those springs with appreciable preloads, exhibit a pronounced increase in spring constant. This behavior suggests that interstitial hardening agents such as carbon and nitrogen were transferred to the spring by the sodium.

I. Examination of Graphite and Cans

Graphite cans were tested for swelling and dimensional stability with respect to irradiation and thermal aging.

1. Can Dimensions

The width, length, and straightness of the three rectangular graphite cans were measured. The measurements showed no significant changes in the dimensions of the cans containing the graphite samples. Comparison of the pre-irradiation measurements with the post-irradiation measurements showed no trends indicating bulging or bowing.

2. Graphite Density

The density of plain and borated graphite was determined on machined blocks sprayed with two coats of acrylic varnish (Spar-Var-Permaclear from Valley Forge Products Co.) to prevent water absorption. The weight of the acrylic coating ranged from 0.3 to 0.5 g per sample (0.54 percent of the weight of the lightest sample) and limited the accuracy of the determinations to ±0.01 g/cm^3. The results are shown in Table XII for all of the SURV subassemblies examined to date. Individual block densities were not determined originally, so it is difficult to determine whether the small density changes noted between SURV-4 and SURV-6 represent the effect of fluence or normal fluctuations in graphite. In any event, these results indicate that no problem due to the swelling of the canned graphite should be anticipated in the EBR-II neutron shield.

3. Gas Release From Cans

Prior to decanning the graphite, the can was connected to a gas analysis apparatus and laser pierced. The gas contents of the graphite cans is presented in Table XIII.

It is clear that a significant amount of thermal outgassing occurred, particularly from the borated graphite. However, additional helium generation due to the accumulated fluence is noted for the capsules containing borated graphite. The total pressure for the worst case is still less than atmospheric. No problem due to this factor is anticipated for the canned graphite in EBR-II neutron shield.

4. Can Material

There was no evidence of graphite sticking to the metal during the decanning of the graphite. The metallography of the can material and/or its tensile properties were determined in previous SURV examinations [1-3]. These measurements were not repeated for SURV-4 or SURV-6 because the graphite and the stainless steel showed no sign of continuing interaction in the earlier examinations. Specimens were taken and stored for possible future examination.

TABLE XII. Effect of EBR-II Exposure on Graphite Density

Exposure	Type Graphite[a]	Density,[c] g/cm^3	Exposure	Type Graphite[a]	Density, g/cm^3
Unirradiated Control	P B	1.59 1.57	SURV-2 (3.5×10^{19})	P B	1.69 1.56
SURV-6 (0)	P B	1.62 1.57	SURV-3 (1.8×10^{20})	P B	1.66 1.58
SURV-1 (1.5×10^{19})[b]	P B	1.65 1.56	SURV-4 (3.3×10^{20})	P B	1.64 1.60

[a]P refers to plain graphite; B refers to borated graphite.

[b]Values in parenthesis are estimated fluences (>0.8 MeV) in n/cm^2.

[c]Estimated accuracy, ±0.01 g/cm^3.

TABLE XIII. Gas Content of Graphite Cans

Property		SURV-4			SURV-6		
		P[a]	PB[a]	B[a]	P	PB	B
Total Gas, (ml STP*)		1.9	29.5	40.7	7.8	11.2	21.5
Calculated Pressure, psia		0.8	9.2	11.8	3.0	3.6	6.1
Composition, Vol. %	He	63	99	77	93	98	30
	N_2[c]	33	0.9	18	6.3	1.8	69
	O_2	0.3	0.01	4.8	0.04	0.02	0.01
	Ar	3	0.01	0.2	0.2	0.03	1.0
	CO_2	0.2	0.01	0.1	0.1	0.03	ND[b]
	H_2	0.2	ND	ND	0.1	0.3	ND

*Standard Temperature and Presssure

[a]P refers to plain graphite loading, B to borated, and PB to a mix of plain and borated.

[b]Not detected.

[c]The presence of significant nitrogen concentration probably indicated in-leakage during the initial vacuum filling and sealing.

IV. DISCUSSION

This report is the first in the series to provide means of separating the effects of fluence from those of prolonged high-temperature exposure. The individual comparisons have been made in each section and both factors have been important. In some instances, such as microstructural change, the influence of the thermal aging has been greater than that of neutron fluence.

The basic material of construction for EBR-II, Type 304 stainless steel, continued to perform well after exposure to 2.2 x 10^{22} n/cm^2 at 370°C. Ductility was still quite good and impact strength was excellent.

Unclad tantalum and Berylco-25 components have been removed from the reactor due to their interaction with the sodium coolant, but no other material has undergone significant corrosive effects.

In most cases, there is no difference in mechanical properties of materials exposed to helium or reactor sodium. However, Inconel X750 springs exposed to reactor sodium showed a marked increase in spring constant.

Graphite densities did not change significantly. Also, there is no evidence of graphite-can interaction, so no problems with the canned graphite neutron shield are anticipated.

ACKNOWLEDGMENTS

We gratefully acknowledge the assistance of F. S. Kirn in the calculation of the fluence, and the technical assistance of D. J. Dorman and J. D. Staffon in performing the density determinations and mechanical property tests and M. T. Laug in analyzing the gases in the graphite cans. Thanks to S. Martinez for valuable assistance in preparing this document.

Work supported by the US Department of Energy under contract No. W-31-109-ENG-38.

REFERENCES

1. S. Greenberg, "The EBR-II Materials-Surveillance Program: I. Program and Results of SURV-1," ANL-7624 (September 1969).

2. S. Greenberg, R. V. Strain, and E. R. Ebersole, "The EBR-II Materials-Surveillance Program: II. Results of SURV-2," ANL-7682 (June 1970).

3. S. Greenberg, R. V. Strain, and E. R. Ebersole, "The EBR-II Materials-Surveillance Program: III. Results of SURV-3," ANL-7937 (September 1972).

Distribution for ANL-98/3

Internal:

T. R. Allen (5)
J. I. Cole
D. C. Crawford
D. J. Hill
G. L. Hofman
R. W. King
J. D. B. Lambert
D. L. Porter
W. E. Ruther (5)
B. G. Storey
R. V. Strain
T. C. Totemeier
H. Tsai
L. C. Walters
TIS Files

External:

DOE-OSTI (2)
ANL-E Library
ANL-W Library
Manager, Chicago Operations Office, DOE