NMI-1233

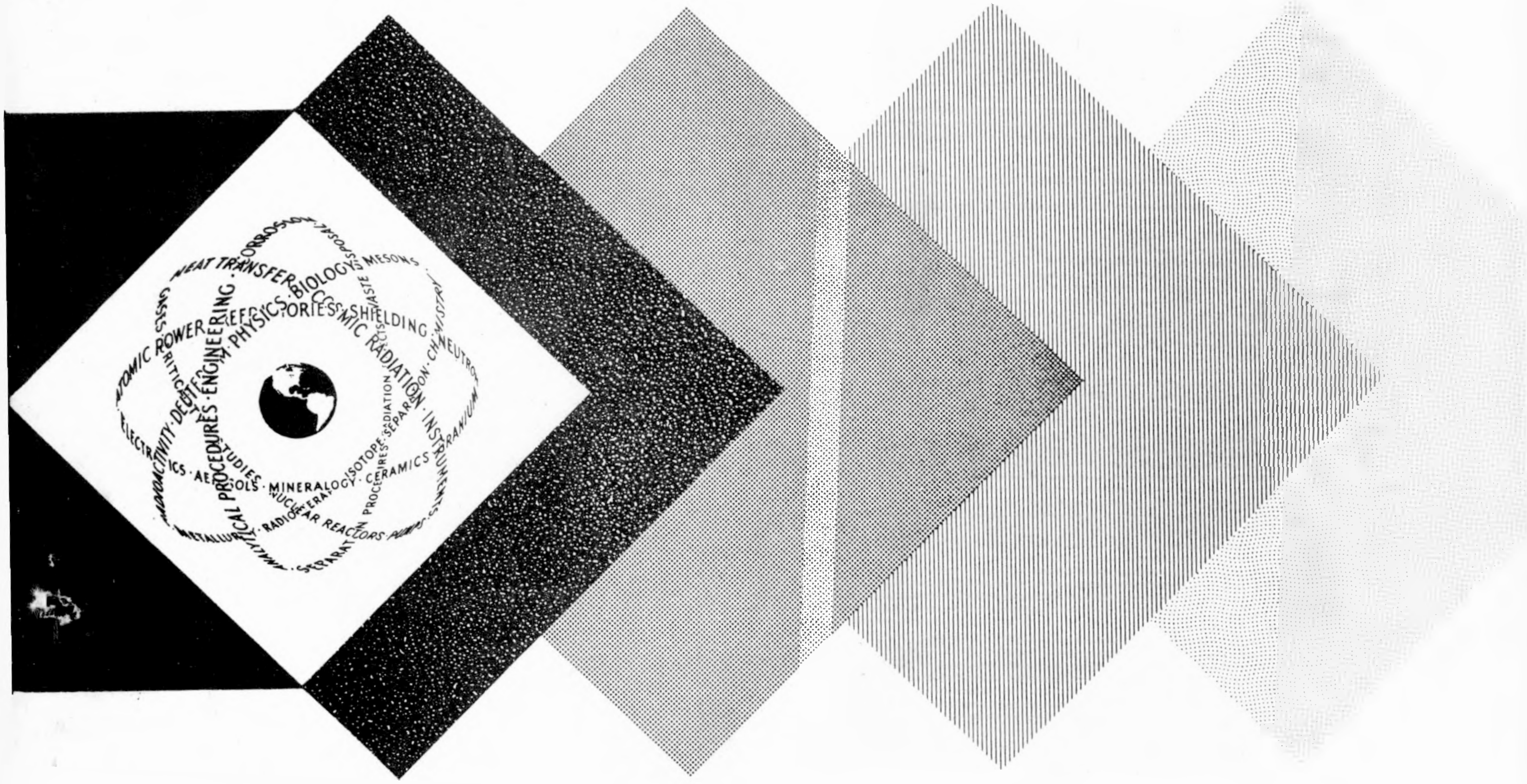
METALLURGY AND CERAMICS

INTERDIFFUSION IN ZIRCALOY-2 CLAD U-2 w/o Zr FUEL MATERIALS AND ITS EFFECT UPON CORROSION BEHAVIOR

By
A. L. Geary

March 24, 1960

Nuclear Metals, Inc.
Concord, Massachusetts



UNITED STATES ATOMIC ENERGY COMMISSION
Technical Information Service

This report has been reproduced directly from the best available copy.

Printed in USA. Price $0.50 Available from the Office of Technical Services, Department of Commerce, Washington 25, D. C.

AEC Technical Information Service Extension
Oak Ridge, Tennessee

NMI-1233

Interdiffusion in Zircaloy-2 Clad U - 2 $^{w}/_{o}$ Zr Fuel Materials and Its Effect Upon Corrosion Behavior

A. L. Geary

March 24, 1960

Nuclear Metals, Inc.
Concord, Massachusetts

Contract AT(30-1)-1565

A. R. Kaufmann
Technical Director

TABLE OF CONTENTS

LIST OF TABLES AND FIGURES

ABSTRACT

Properties of the interdiffusion zone in Zircaloy-2 clad U - 2 $^{w}/o$ Zr fuel materials have been investigated and the results correlated with the corrosion behavior of defected specimens in high-temperature water. Electron microprobe and metallographic examinations of the zones were carried out, as well as corrosion studies on specimens annealed after the diffusion treatment. The results indicate that the beneficial effect of the diffusion treatment upon the corrosion behavior of defected specimens results from improved strength properties of the bond zone. During corrosion, the combined strengths of the zone and Zircaloy-2 cladding are great enough to compact the oxide corrosion product at the base of the defect, the compacted oxide inhibiting further attack. The formation of equilibrium alpha uranium in the inner portion of the zone must, however, be minimized in order to achieve the effect of the diffusion treatment.

I. INTRODUCTION

Zircaloy-2 cladding of fuel elements used in water-cooled reactors does not provide complete assurance that corrosion of the uranium or uranium-base alloy core will be prevented. Although the Zircaloy-2 is highly resistant, defects extending through the clad may develop and expose the core to the corrosive action of the high-temperature water. Stresses resulting from conversion of uranium to the less dense oxide deform and eventually rupture the cladding around the defect. Progressive exposure of the core by failure of the cladding can ultimately lead to complete destruction of the fuel element.

Diffusion heat treatments at temperatures above 800°C (1470°F) neutralize defects in the Zircaloy-2 cladding when the core is U - 2 $^{w}/o$ Zr. The treatments are not effective, however, when the core is unalloyed uranium or thorium.(1) The beneficial effect of the diffusion treatments was first established by Lamartin and Levine(2), who found that diffusion-treated specimens containing 7-mil defects, which otherwise would have failed in less than 4 hours, withstood exposures to 316°C (600°F) water well in excess of 1000 hours. Metallographic examination of the corroded specimens indicated that attack of the core at the base of the defect started but did not continue. The net effect appeared to be compaction of a small pocket of corrosion product with little, if any, deformation of the Zircaloy-2 clad.

Jenkins(3) and Isserow(4), extending the work of Lamartin and Levine, demonstrated that there is no unique temperature range for effective diffusion treatment. Treatments at various temperatures will impart corrosion resistance provided that time is allowed for the necessary diffusion to occur, the necessary diffusion depending upon factors such as cladding thickness and depth, and diameter of the defect. To be effective, however, interdiffusion must be followed by rapid cooling. Specimens furnace-cooled or interrupted-quenched at 690°C (1275°F) and 600°C (1110°F) failed as rapidly as the extruded specimens. Isothermal holds in the vicinity of 600°C appeared to be the more detrimental.

The corrosion resistance exhibited by defected specimens after diffusion treatment has been attributed to confinement and compaction of the oxide corrosion product at the base of the defect, the compacted oxide inhibiting further attack of the core. According to this view, U-Zr alloys formed during the treatment are strong enough to resist deformation by the stresses resulting from conversion of the metal to the less dense oxide, and compaction of the oxide results. Evidence which forms the basis for this conclusion has been summarized by Isserow and Jenkins.(1)

An attempt to demonstrate that compaction of the oxide inhibits attack did not yield conclusive results.(3) A 7- to 8-mil diameter unalloyed uranium core clad with 0.15-inch Zircaloy-2 corroded far into the rod away from the end exposed to high-temperature water or steam. The diameter of the UO_2 formed was approximately 4 mils greater than that of the uncorroded uranium. Thus, at least for unalloyed uranium, corrosion inhibition by self-compaction of the oxide appears unlikely. The corrosion behavior and density changes of unalloyed uranium differ from those of U - 2 $^w/o$ Zr so that the results may not be indicative of those which would be obtained with the alloy core.

Although these studies indicated the conditions required to establish corrosion resistance in defected specimens, they did not provide a complete understanding of the phenomena. The purpose of this work, therefore, was to examine the properties of the interdiffusion zone and to correlate these properties with the corrosion behavior of the defected specimens.

II. EXPERIMENTAL

The 3/8-inch diameter rods which provided stock material for specimens used in this work were produced by coextrusion of composite billets at 650°C (1200°F).(5) U-Zr alloys containing 2 to 15 $^w/o$ Zr clad with 15- and 30-mil Zircaloy-2 were produced. The strength of the core-clad bond in each of the extruded rods was determined at several positions by the "chisel test." In this test, thin discs were split along a diameter and examined for separation at the core-clad interface.

A. Corrosion Studies

Procedures established by Jenkins(3) were followed throughout the preparation of corrosion specimens containing the 7-mil defects. Two-inch lengths of the 15-mil Zircaloy-2 clad U - 2 $^w/o$ Zr core rods were fitted with leak-tight

Zircaloy end closures, and four 7-mil defects, located 90 degrees apart around the circumference at the mid-length of the specimen and extending through the clad, were inserted by an "automatic" drilling technique.

Corrosion specimens of U-Zr binary alloys containing 2, 5, 10 and 15 $^{w}/o$ Zr were obtained by machining the Zircaloy-2 from 1-inch sections of the extruded rods with these core compositions. The diameter of the machined specimens was 0.33 inch.

Prior to heat treatment the corrosion specimens were thoroughly degreased with trichlorethylene, wrapped with 1-mil tantalum foil, and sealed in evacuated Vycor capsules. All specimens except those tested in the as-extruded condition were interdiffused 7 hours at 880°C (1615°F) and either water-quenched or furnace-cooled at approximately 5°C/min. Since the water quench was carried out without breaking the Vycor tube, it was not a drastic quench and the specimens retained color for 1 to 2 minutes. Following the water quench, selected specimens were isothermally treated for times up to 2 hours at 500° to 715°C (930° to 1320°F) and water-quenched.

Corrosion tests in the high-temperature water were carried out in static autoclave equipment. High-purity water with a resistivity greater than 0.5 megohm-cm and a pH of 6.7 at room temperature was used. Specimens tested to failure or to total exposures of 1000 hours, whichever occurred first, were corroded in 343°C (650°F) water. Those for metallographic examination were corroded in 277°C (530°F), since the lower corrosivity of the water at this temperature permitted greater control of the extent of corrosion.

The unclad U-Zr binary alloys were corroded one hour at 299°C (570°F). The weight losses determined in these tests, however, included the losses that occurred during the approximately 2 hours required to heat the autoclave assembly to the test temperature.

B. Bond Strength Tests

Bond strength test specimens were machined from 1-inch long sections of the 30-mil clad U - 2 $^{w}/o$ Zr extruded rod heat treated in the same manner as the corrosion specimens. The cladding was removed from one-half of the specimen and the core from the other half, leaving a 0.030-inch length of the bond zone that could be tested in shear. The specimens after test were examined metallographically to determine the location of the fracture.

C. Electron Probe and Metallographic Examination of Diffusion Zone

Specimens of U - 2 $^{w}/o$ Zr clad with 15- and 30-mil Zircaloy-2 were prepared for metallographic examination and chemical analysis of the diffusion zone. These specimens were defected and heat treated in the same manner as the defected corrosion specimens. Metallographic examination at the cross section of the defect was carried out by standard techniques after electropolishing in an acetic-perchloric solution.

Compositions in the diffusion zone were analyzed in 0.5-mil steps by the electron microbeam probe technique developed by Castaign.(6) A beam of constant energy electrons was focussed onto the specimen in a spot about 5 microns in diameter and the intensities of the uranium L-alpha I and zirconium K-alpha irradiations measured with a suitable counter tube. Comparison of these intensities with those from standard samples yielded the composition of the unknown.

III. RESULTS

A. Corrosion

1. Specimens Containing 7-mil Defects

Results of the corrosion tests on specimens containing 7-mil defects are given in Table I. These specimens consisted of a U - 2 $^{w}/o$ Zr core clad with 15-mil Zircaloy-2. The 7-hour diffusion treatment at 880°C, when followed by air cooling, effectively established corrosion resistance in this material, treated specimens surviving exposures to 343°C water in excess of 1000 hours. The diffusion treatment is, however, not effective when followed by furnace cooling. The furnace-cooled specimens failed after 3 to 4 hours' exposure, approximately the same time as that required to cause failure of the untreated (as-extruded) specimens.

Corrosion resistance of the diffusion-treated and water-quenched specimens is also destroyed by isothermal annealing in the range 550° to 715°C. However, the minimum annealing times required to cause failure vary with temperature, shorter times being required at 600° and 690°C than at either 650°C or temperatures below 550°C.

The corrosion resistance of the furnace-cooled or annealed specimens can be restored by an additional treatment for 2 hours at 880°C followed by a water quench. Specimens receiving this treatment exhibit corrosion resistance comparable to that of the diffusion-treated and water-quenched specimens.

Photomicrographs of the corroded areas at the base of the defects are shown in Fig. 1. Three different types of attack can be seen in this Figure. In the as-extruded specimen, preferential attack of the core takes place in the area along the core-clad interface, the remainder of the exposed core corroding uniformly. This preferential attack becomes more extensive as the exposure time is increased. In addition to preferential attack of the core, corrosion of the Zircaloy-2 clad occurs in areas where the core has been removed. Measurements indicate that 1 to 2 mils has corroded during the 1-hour exposure to the 277°C water.

The furnace-cooled specimen, Fig. 1b, exhibits preferential attack of the diffusion zone in the areas exposed by corrosion of the core but not of the core itself. This attack is highly localized and appears as small "fingers" of corrosion penetrating upward into the zone from the core. One of these "fingers" is shown at 150X magnification in Fig. 2. Transformation of a portion of the diffusion zone immediately above the corrosion pocket is also evident in Fig. 1b. The nature of this transformation, however, has not as yet been determined.

Attack in the specimens annealed after the diffusion treatment is similar to that in the furnace-cooled specimen. Specimens annealed 2 hours at 600°, 650°, and 690°C exhibit localized attack of the diffusion zone and transformation of a portion of the diffusion zone immediately above the corrosion pocket. Because of their similarity to the furnace-cooled specimen, photomicrographs of the defect areas in the annealed specimens are not shown.

The attack in the specimens discussed above should be compared with that in the diffusion-treated specimen, Fig. 1c, which withstood exposure to 343°C water for 900 hours without failure. In the latter, corrosion is characterized by the formation of a spherical corrosion pocket without preferential attack of either the core or the diffusion zone. The slight corrosion of the zone that occurs does so laterally from the wall of the defect. In addition, there is no evidence that portions of the zone transformed during the corrosion.

2. U-Zr Binary Alloys

The U-Zr binary alloys containing up to 15 $^{w}/o$ Zr corroded rapidly in high-temperature water. Weight losses of the alloys after exposure to 299°C for one hour are given in Table II. There is, however, an effect of zirconium content upon corrosion; alloys containing 2 and 5 $^{w}/o$ Zr corrode more rapidly than those containing 10 and 15 $^{w}/o$.

Corrosion of these alloys appears to be independent of thermal history. Variations in the weight losses after different heat treatments are well within the reproducibility of these tests.

B. Bond Strength

Results of the bond strength tests were inconclusive. Each of the specimens fractured in the Zircaloy-2 clad rather than at the core-clad bond. Evidently, the strength of the bond is greater than that of the Zircaloy-2 clad, and this superiority is not affected by any of the thermal treatments.

C. Electron Microprobe Analysis of the Interdiffusion Zone

Compositions within the bond layer of the as-extruded specimen could not be determined by the electron microprobe technique. Successive analyses, 0.5 mil apart, traversing the bond area, showed only an abrupt change in composition from that of the core to that of the clad. The short time at the extrusion temperature apparently limits diffusion to distances of less than 0.5 mil.

Compositions within the zone of specimens interdiffused at 880°C for 7 and 24 hours could, however, be readily determined. In these specimens, the zone was 6 to 8 mils wide so that, when traversing the zone, 10 to 15 individual analyses could be made. Results of these analyses are shown in Fig. 3, where zirconium content is plotted as a function of the distance from the original core-clad interface.

The curves in Fig. 3 show that at 880°C uranium diffuses more rapidly than zirconium. Penetration of uranium into the clad is 2.5 to 3 times that of zirconium into the core. The greater mobility of uranium in this system has been reported previously by Lamartin and Levine,(2) and in the U-Zr system by Adda and co-workers(7). However, a quantitative comparison cannot be made because of the lack of a suitable solution of Fick's Law for the cylindrical specimens.

Cladding thicknesses in the range of 15 to 30 mils apparently do not alter the diffusion boundary conditions. The concentration-penetration curve for the 30-mil clad specimen, also shown in Fig. 3, agrees with that of the 15-mil clad specimen. Misfit between the two curves occurs principally in the region where the concentration changes rapidly with distance. It is also in this region that errors in the distance measurements would have the greatest effect upon the analyses.

The gradient in specimens diffused 7 hours at 880°C is not changed significantly by subsequent heat treatments of 2 hours at 600°C or 2 hours hours at 600°C followed by 2 hours at 880°C. As shown in Fig. 4, the concentration-penetration curves are in reasonable agreement. Discrepancies among the curves occur, as in the comparison of the 15- and 30-mil clad specimens, in the region where the composition changes rapidly with distance.

Electron microprobe analyses were also carried out in the area at the base of the defect. The surface and probe trace in these analyses should ideally have coincided with the defect axis. These conditions could not be achieved experimentally, although precautions were taken during specimen preparation and during the analyses. The results obtained were thus influenced by a small displacement of the surface and probe trace from the defect axis.

The results, Table III, show that the presence of the defect limits diffusion of zirconium into the area beneath it. Zirconium penetrates less than 0.5 mil into the core during the 7-hour diffusion treatment, compared with about 1.5 mils in the diffusion zone. Because of the limited diffusion, the maximum zirconium content of the material beneath the defect does not exceed 10 $^{w}/o$ and may be less than 5 $^{w}/o$. Uranium appears to diffuse more rapidly; at equal distances from the original interface, the uranium content in the clad near the defect is significantly higher than in the zone. These higher uranium contents may result from surface diffusion at the tip of the defect where the core is exposed.

D. Microstructure of the Diffusion Zone

Microstructures of the diffusion zones in the water-quenched, furnace-cooled, and 600° and 690°C annealed specimens are illustrated, together with the zirconium gradient in the zone, in Fig. 5. The specimens annealed at 550° and 650°C are not shown, since their microstructures are similar to those of the 600° and 690°C annealed specimens, respectively.

The photomicrographs show that the gamma phase in much of the diffusion zone transforms partially or completely to produce a complex series of structures. In addition, the structures are further complicated by transformation during the 600° and 690°C annealing treatments. However, if the zone is divided into layers with the following zirconium contents, 2 to 25, 25 to 80, and 80 to 98 $^{w}/o$ Zr, the structures are found to resemble those of U-Zr binary alloys with similar zirconium contents and similar thermal histories. Bauer(8) and Chubb(9) have described the transformation kinetics and microstructures of the binary alloys, so they will not be repeated here. The following discussion will be limited to the microstructural differences which may have an effect upon corrosion behavior of the defected specimens.

Compared with the water-quenched specimen, the furnace-cooled and the annealed specimens are characterized by a more extensive transformation in each layer of the diffusion zone. This is not immediately apparent for the 2 to 25 w/o Zr layers since their structures are not resolved in the photomicrographs. These layers etched more rapidly than the remainder of the zone and could not be brought into focus for the photograph. Visual examination indicated that this layer in the water-quenched specimen consisted of fine acicular alpha-uranium, apparently formed by nucleation and growth from the gamma. The acicular alpha-uranium structure was also found in the furnace-cooled specimen and in those annealed at 600° and 690°C, but in each the alpha platelets were larger than those in the water-quenched specimen. The alpha platelet size increased in the following specimen order: 600°C annealed, 690°C annealed, and furnace-cooled.

The microstructures of the 80 to 98 w/o Zr layer closely resemble those of the 2 to 25 w/o layer, although alpha-zirconium rather than alpha-uranium is formed. The finest structure occurs in the water-quenched specimen, and the structure becomes progressively coarser in the 600°C annealed, 690°C annealed, and furnace-cooled specimens.

Because of the numerous phases which may be present (retained gamma, non-equilibrium epsilon, as well as equilibrium epsilon, alpha-uranium and alpha-zirconium), differences in the microstructures of the layer containing 25 to 80 w/o Zr cannot be described in detail. X-ray analyses of this layer would be required to identify the phases in each of the structures. However, comparison of the photomicrographs indicates that the structure in the furnace-cooled specimen and in the annealed specimens is more complex than that in the water-quenched specimen, suggesting that more extensive transformations have occurred in the former.

IV. DISCUSSION OF RESULTS

The results of this work demonstrate, once again, the beneficial effect of a diffusion treatment upon the corrosion behavior of defected specimens. The beneficial effect is, however, observed only in specimens rapidly cooled (water-quenched or air-cooled) from the treatment temperature; these specimens withstand exposures in 343°C water of up to 1000 hours without deformation or failure of the cladding around the defect. On the other hand, specimens furnace-cooled or those water-quenched and then annealed at 550°- 720°C for times greater than about 0.25 hour fail as rapidly as the untreated specimens.

These results are in agreement with those reported by Isserow(4), with the exception that the isothermal annealing times which lead to corrosion failure are shorter. Isserow found that, for specimens interrupted-quenched after interdiffusion, isothermal holds at 600° and 690°C in excess of one hour were required to cause failure. The reasons for this dependence of corrosion behavior upon quenching technique have not as yet been investigated.

The photomicrographs of the corroded specimens demonstrate that attack occurred at the base of the defects in all specimens; even the specimen exposed 900 hours in the 343°C water exhibits a pocket of corrosion product. Thus the beneficial effect of the diffusion treatment cannot be attributed to the formation of a corrosion-resistant alloy at the base of the defect. This conclusion is further substantiated by the electron microprobe analyses and the corrosion results obtained for the low-zirconium alloys. The microprobe analyses indicate that zirconium content at the base of the defect probably does not exceed 10 w/o, a composition that corrodes rapidly in high-temperature water.

The photomicrographs of the corroded specimens also indicate that failure of the furnace-cooled and the annealed specimens results from corrosion of the inner layer of the zone followed by localized attack of the outer layer. Since this corrosion reduces the effective cross-section of the zone and provides sites of high stress concentration, the combined strengths of the zone and cladding are no longer great enough to cause compaction of the oxide. In contrast, compaction of the oxide, and presumably corrosion inhibition, occurs in the rapidly-cooled specimen without exposure of the outer portion of the zone except at the wall of the defect.

Whether compaction of the oxide occurs before exposure and localized attack of the outer portion of the zone is apparently related to the amount and distribution of equilibrium alpha-uranium in the inner portion. The differences in the zirconium concentration gradients do not seem large enough to account for this behavior. Further, the amount and distribution of equilibrium alpha is known to affect the corrosion of U-Zr binary alloys.(10) Alloys containing up to 20 $^{w}/o$ U are more resistant in the gamma-quenched than in the alpha-annealed condition. Although the amount cannot be measured quantitatively, the photomicrographs of the uncorroded specimens indicated that the furnace-cooled and the annealed specimens contain more equilibrium alpha than the rapidly-cooled specimen. There may, however, be a critical amount of equilibrium alpha that can be tolerated without failure, since the air-cooled specimens and those annealed for short times, undoubtedly containing greater amounts of this phase, are as resistant as the water-quenched specimens.

Penetration of the outer portion of the zones in the furnace-cooled and in the annealed specimens is much more rapid than would be predicted from corrosion rates reported for the epsilon- and alpha-zirconium phases. In 343°C water, epsilon corrodes at about 0.2 $mg/cm^2/hr$ and alpha-zirconium at about 400 $mg/cm^2/t^{1/3}$, where t is the time in days. The reasons for the accelerated attack have not been investigated. The photomicrograph shown in Fig. 2 suggests, however, that it may be due to corrosion product hydrogen adsorbed by the metal or to high stresses developed at the tip of the penetration.

The interdiffusion zone of the as-extruded specimen, in contrast to that of the diffusion-treated specimens, appears to have little, if any, effect upon corrosion within the defect. Apparently this layer, because of its limited thickness, possesses neither the corrosion resistance nor the strength properties required for compaction of the oxide.

V. TABLES AND FIGURES

TABLE I

Corrosion of Defected Zircaloy-2 Clad U - 2 $^{w}/o$ Zr Core Specimens in 343°C (650°F) Water

All specimens, encapsulated in evacuated Vycor, contained 7-mil diameter defects extending into core. Initial heat treatment at 880°C for 7 hours.

Specimen No.	Cooling and Subsequent Heat Treatments	Corrosion	
		Time (hrs)	Remarks
1	air-cooled	1149	no failure
2	" "	1149	no failure
3	furnace-cooled	1	failed at 4 defects
4	" " ; 2 hrs at 880°C; water-quenched	923	no failure
5	water-quenched; 0.25 hr at 715°C; " "	4	failed at 4 defects
6	" " ; 2 hrs at 690°C; " "	4	failed at 4 defects
7	" " ; 1 hr at 690°C; " "	4	failed at 4 defects
8	" " ; 0.5 hr at 690°C; " "	4	failed at 4 defects
9	" " ; 0.25 hr at 690°C; " "	4	failed at 4 defects
10	" " ; 0.17 hr at 690°C; " "	1036	no failure
11	" " ; 2 hrs at 650°C; " "	3	failed at 4 defects
12	" " ; 1 hr at 650°C; " "	4	failed at 2 defects
13	" " ; 0.5 hr at 650°C; " "	23	failed at 2 defects
14	" " ; 0.25 hr at 650°C; " "	487	failed at 1 defect
15	" " ; 2 hrs at 600°C; " "	1	failed at 4 defects
16	" " ; 1 hr at 600°C; " "	4	failed at 4 defects
17	" " ; 0.5 hr at 600°C; " "	4	failed at 2 defects
18	" " ; 0.25 hr at 600°C; " "	4	failed at 2 defects
19	" " ; 0.17 hr at 600°C; " "	1036	no failure
20	" " ; 2 hrs at 600°C; " " 2 hrs at 880°C; " "	771	failed at welds; no failure at defects

(Table continued on next page)

TABLE I (cont'd.)

Specimen No.	Cooling and Subsequent Heat Treatments	Corrosion	
		Time (hrs)	Remarks
21	water-quenched, 1 hr at 550°C; water-quenched	4	failed at 2 defects
22	" " ; 0.5 hr at 550°C; " "	4	failed at 2 defects
23	" " ; 0.25 hr at 550°C; " "	1047	no failure
24	" " ; 0.5 hr at 500°C; " "	1036	no failure

TABLE II

Corrosion of Uranium-Zirconium Binary Alloys Containing Up to 15 $^{w}/o$ Zr

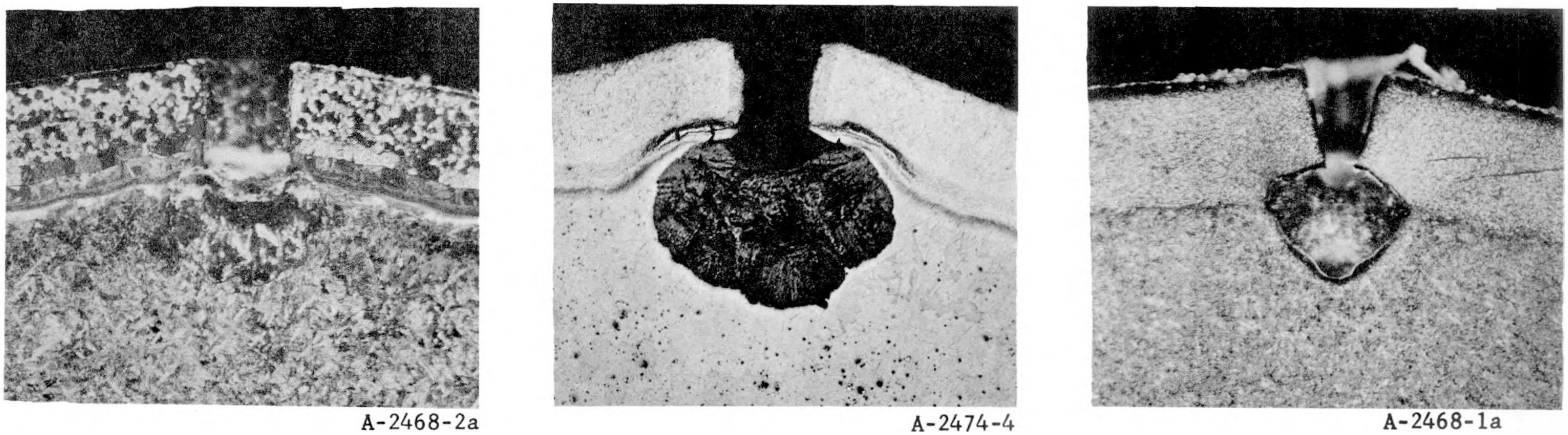
(corroded one hour in 299°C [570°F] water)

Condition	Weight Loss (gm/cm^2)			
	2 $^{w}/o$ Zr	5 $^{w}/o$ Zr	10 $^{w}/o$ Zr	15 $^{w}/o$ Zr
As extruded	2.35	--	--	--
7 hrs at 880°C and water-quenched	1.98	2.05	1.49	1.18
7 hrs at 880°C and furnace-cooled	2.07	1.85	1.43	1.22
7 hrs at 880°C and water-quenched plus 2 hrs at 690°C and water-quenched	--	2.06	1.32	1.16
7 hrs at 880°C and water-quenched plus 2 hrs at 650°C and water-quenched	--	1.91	1.59	1.04
7 hrs at 880°C and water-quenched plus 2 hrs at 600°C and water-quenched	2.33	2.15	1.59	1.14

TABLE III

Zirconium Content at the Base of Defects in 15-mil Clad Specimens

	No. 26	No. 27	No. 28
Distance from original interface (mils)	7 hrs at 880°C $^{w}/_{o}$	7 hrs at 880°C and 2 hrs at 600°C $^{w}/_{o}$	7 hrs at 880°C; 2 hrs at 600°C; 2 hrs at 880°C $^{w}/_{o}$
-2.0	2	2	2
-1.5	--	--	--
-1.0	2	2	2
-0.5	--	--	2
0	6	5	5
+0.5	6	--	3
+1.0	4	5	7
+1.5	--	--	8

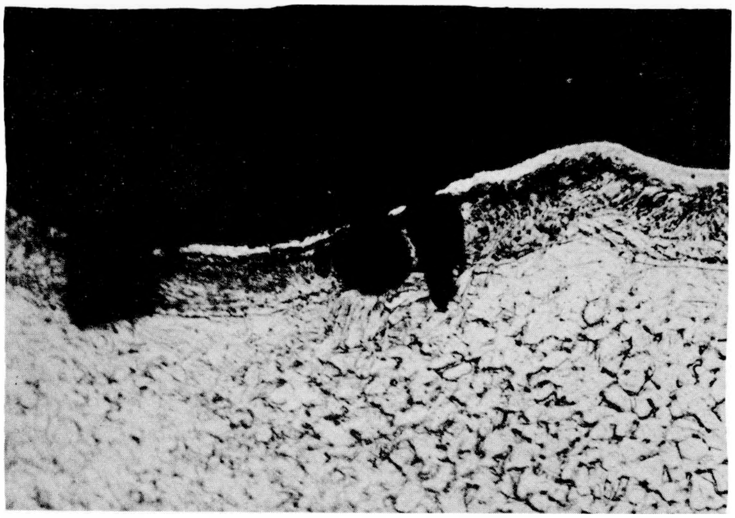


1a. Untreated (as-extruded) specimen corroded 1 hr in 277°C water. Electropolished, 50X.

1b. Specimen diffusion treated 7 hrs at 880°C and furnace cooled. Corroded 4 hrs in 277°C water. Mechanically polished, 50X.

1c. Specimen diffusion treated 7 hrs at 880°C and water quenched. Corroded 900 hrs in 343°C water. Electropolished, 50X.

Fig. 1. Sections through defects of specimens corroded in high-temperature water.



A-2474-4a (150X Bt)

Fig. 2. Localized corrosion of interdiffusion zone. Specimen interdiffused 7 hrs at 880°C and annealed 3 hrs at 690°C. Corroded 5 hrs in 277°C water.

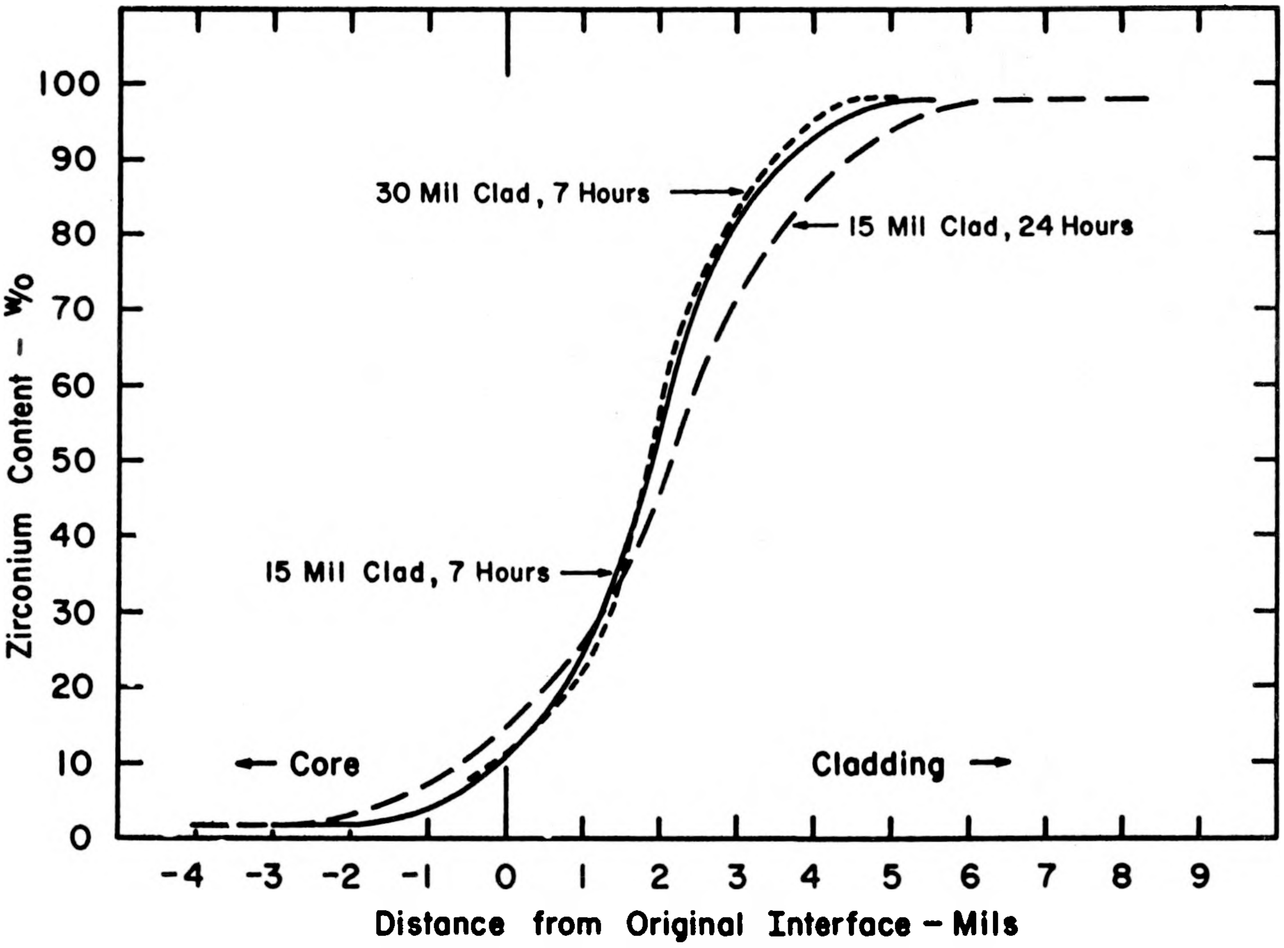


Fig. 3. Concentration-penetration curves for specimens interdiffused at 880°C.

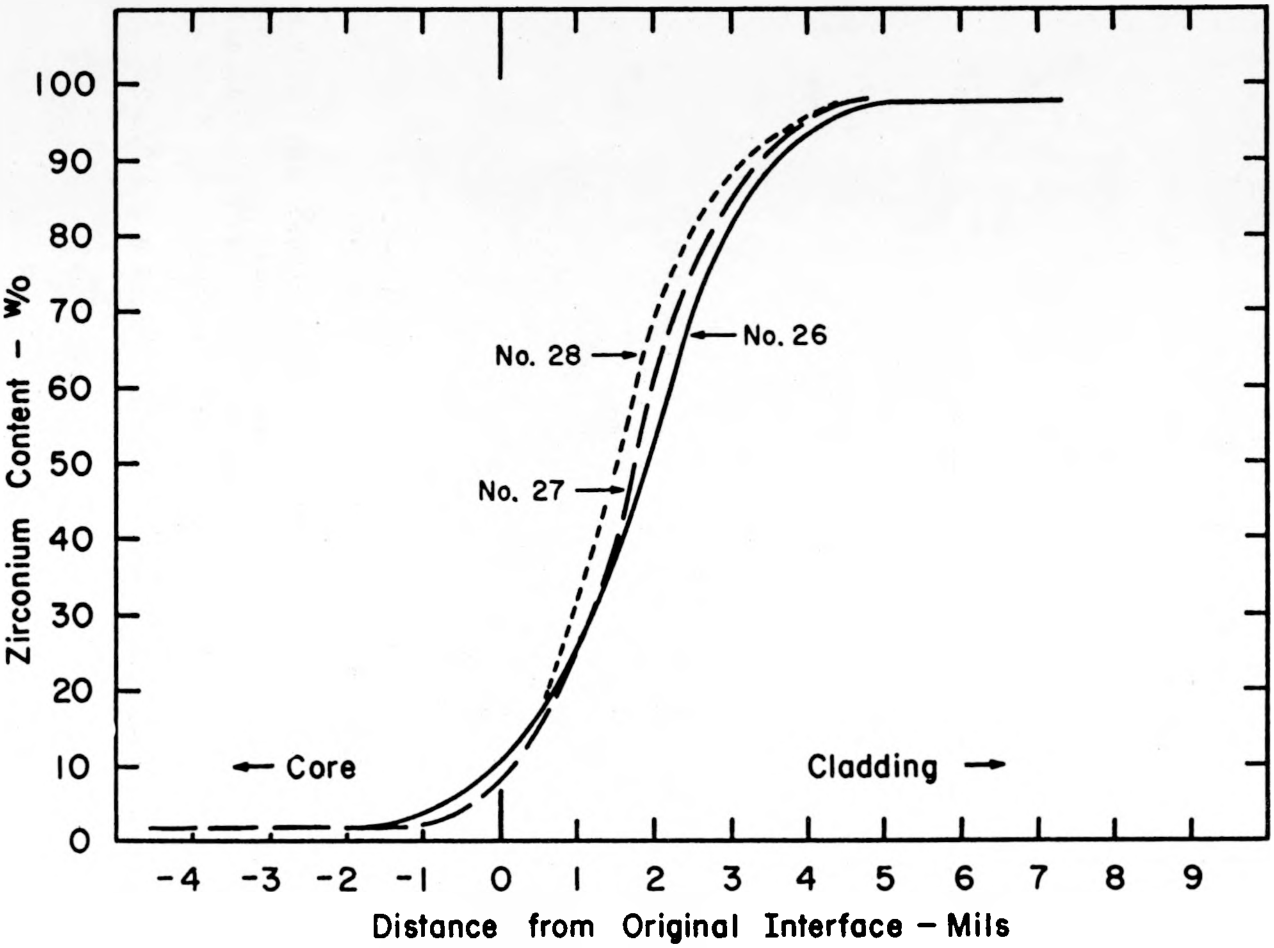
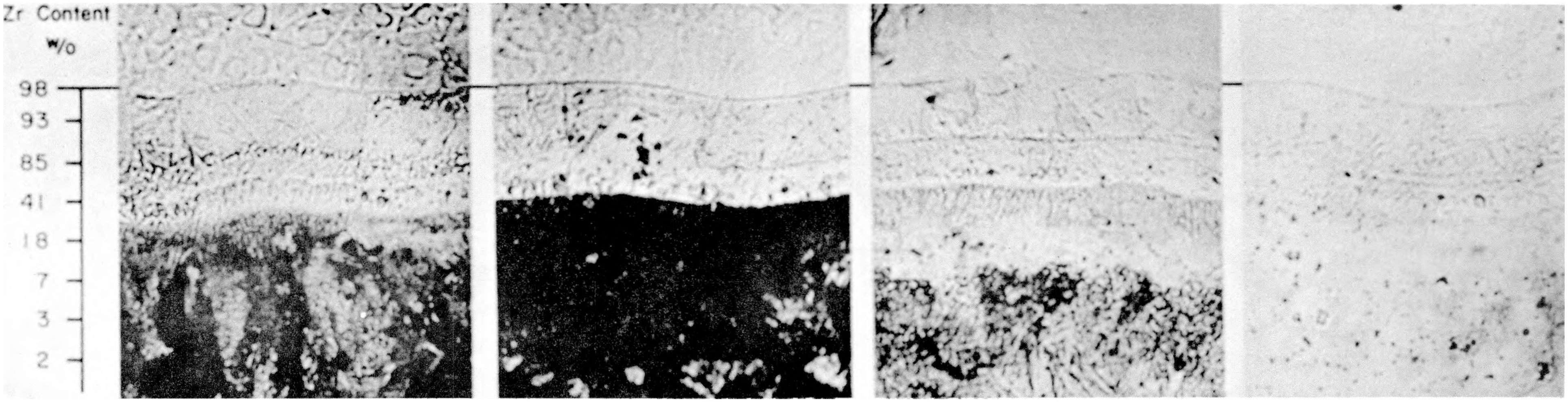


Fig. 4. Concentration-penetration curves for 15-mil clad specimens.

No. 26 - 7 hrs at 880°C.

No. 27 - 7 hrs at 880°C and 2 hrs at 600°C.

No. 28 - 7 hrs at 880°C, 2 hrs at 600°C and 2 hrs at 880°C.



A-2410-1b
250X, Bt.

A-2410-2b
250X, Bt.

A-2424-3b
250X, Bt.

A-2424-1b
250X, Bt.

(a) Diffused 7 hr at 880°C and water quenched.

(b) Diffused 7 hr at 880°C and furnace cooled.

(c) Diffused 7 hr at 880°C and water quenched. Annealed 2 hr at 690°C and water quenched.

(d) Diffused 7 hr at 880°C and water quenched. Annealed 2 hr at 600°C and water quenched.

<u>Fig. 5</u> - Interdiffusion zones of thermally-treated Zircaloy-2 clad U - 2 $^{w}/o$ Zr.

V. REFERENCES

1. S. Isserow and R. G. Jenkins, Aqueous Corrosion of Zircaloy-clad Fuel Elements with High Uranium Cores, NMI-TJ-23, Sept. 20, 1959.

2. F. H. Lamartin, Jr. and S. Levine, Corrosion Prevention in Clad Nuclear Fuel Rods, S.M. Thesis, Massachusetts Institute of Technology (1955).

3. R. G. Jenkins, The Diffusion Anneal for Increasing the Effectiveness of Zircaloy Cladding, NMI-1217, to be published.

4. S. Isserow, Corrosion Behavior of Defected Fuel Elements with U - 2 $^{w}/o$ Zr Core Clad with Zircaloy-2, NMI-4364, July 21, 1958.

5. A. R. Kaufmann, J. L. Klein, P. Loewenstein, H. F. Sawyer, "Zirconium Cladding of Uranium and Uranium Alloys by Coextrusion," Fuel Elements Conference, Paris, November 18-23, 1957 (TID-7546).

6. R. Castaign, J. Philibert, C. Cussard, "Electron Probe Microanalyzer and its Application to Ferrous Metallurgy," Trans. AIME, 209, 389 (1957).

7. Y. Adda, J. Philibert, H. Faraggi, "Investigation of the Interdiffusion Phenomena in the System Uranium Zirconium," Revue de Metallurgie, 54, 597 (1957).

8. A. A. Bauer, "Kinetics of Transformation of Zirconium-uranium Alloys," p. 15, BMI-1350 (1959).

9. W. Chubb, "The Effect of Heat Treatment on the Microstructure of Zirconium-uranium Alloys," p. 43, BMI-1350 (1959).

10. W. E. Berry, "The Corrosion Behavior of Zirconium-uranium Alloys," p. 93, BMI-1350 (1959).