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ELECTRON METALLOGRAPHY OF THE Al-Ni-U BOND

by

C. L. Angerman

Pile Materials Division

December 1957

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Explosives Department - Atomic Energy Division
Technical Division - Savannah River Laboratory

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ABSTRACT

The diffusion zones formed during the bonding of nickel-plated uranium cores to aluminum sheaths were identified by correlating the appearance of these zones with those identified in diffusion couples. A relationship between the structure and the strength of the bond was observed.

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LIST OF FIGURES

Some of the detail in the photomicrographs was lost during reproduction by halftone. Photomicrographs are available from the Savannah River Laboratory.

ELECTRON METALLOGRAPHY OF THE Al-Ni-U BOND

INTRODUCTION

Because nickel is being used as a diffusion barrier between the uranium core and the aluminum sheath of a variety of fuel elements[1,2], it is of interest to know the composition of the diffusion zones formed during bonding and the relationship between the microstructure of the zones and the strength of the bond. This knowledge is particularly useful in the development of fabrication methods for fuel elements. This report describes an investigation of the diffusion zones of the aluminum-nickel-uranium system. Both optical and electron metallography and X-ray diffraction were used to investigate bonded fuel elements and specially prepared diffusion couples.

SUMMARY

There are four intermetallic compounds known in the aluminum-nickel system, and seven intermetallics in the uranium-nickel system; three of the aluminum-nickel intermetallics and all of the uranium-nickel compounds were observed metallographically in diffusion couples. Two of the four zones observed in aluminum-nickel diffusion couples and six of the seven zones observed in uranium-nickel couples were identified by X-ray diffraction. The identities of the remaining zones were deduced from a knowledge of the phase diagram. The zones observed in as-bonded fuel elements were identified by correlating the appearances of the zones with those of the zones identified in the diffusion couples.

In as-bonded fuel elements two aluminum-nickel and two uranium-nickel diffusion zones were observed. They were identified as Al_3Ni and Al_3Ni_2, and U_6Ni and UNi_5, respectively. Uranium dioxide was also found either as a second phase in the UNi_5 layer or as a continuous layer between the U_6Ni and the UNi_5 zones. The oxide particles which occurred as a dispersed second phase coalesced into a continuous layer and finally dissolved during heat treatment.

A qualitative relationship was observed between the presence of a continuous UO_2 layer and the strength of the bond for step-pressed plate elements and for tubular elements that were bonded by the fluid-pressure process. The continuous UO_2 layer was observed in elements that had an average bond strength below 10,000 psi, whereas dispersed particles of UO_2 were observed in the UNi_5 layer of elements that had an average bond strength in excess of 10,000 psi.

DISCUSSION

BACKGROUND

Considerable data have been published on the kinetics of diffusion[3] and X-ray identification[4] of the compounds in the aluminum-nickel system. Recently, Storcheim[5], Castleman[6], and Kidson[7] have studied the solid-state bonding of aluminum to nickel and reported diffusion data and identification of the intermetallics formed.

Relatively little information has been published on the uranium-nickel system. Two disparate phase diagrams have been reported[8,9] and the intermetallics have been partially identified[10]. Kittel[11] has studied the kinetics of diffusion, as has Storcheim[12] in his work on solid-state bonding. No data, though, have been reported on the identification of the diffusion zones formed during bonding.

MATERIALS

The specimens from bonded fuel elements that were used in this study were cut from step-pressed plate elements[1] and from fluid-pressure bonded tubular elements[2]. The results of the study should also apply to any other form of bonding, such as extrusion cladding.

Diffusion couples were made of discs, 3/4 inch in diameter, cut from rolled metal. The uranium was production-quality metal and the aluminum was Type 1100. The nickel was an electroplating anode that had been hot rolled. All discs were approximately 0.2 inch thick.

EXPERIMENTAL TECHNIQUES

Diffusion Couples

As an aid in the interpretation of the bond structures of clad fuel elements, diffusion couples of aluminum and nickel, and of uranium and nickel were fabricated in a modified metallographic mounting press. A nichrome coil that was capable of heating to 565°C was wound around the sleeve of the mounting die. The nichrome was insulated from the steel sleeve with mica and was held in place with "Alundum" cement. The temperature was measured with a chromel-alumel couple that was located 1/8 inch from the diffusion couple.

The surfaces of the diffusion couple components were prepared to ensure good contact. The mating surfaces were ground on 320-grit paper and the uranium portion of the couple was etched in nitric acid at room temperature prior to insertion into the press. A pressure of 8,000 psi was applied to the couple before heating to minimize oxidation. The couples were held under pressure for five minutes at a temperature of 565°C.

Diffusion Anneals

Long-term anneals were made in an air furnace with the specimens sealed in evacuated "Vycor" tubes. The tubes were evacuated to a pressure of at least 1×10^{-4} mm of mercury. For further protection against oxidation, the couples were wrapped in tantalum foil before they were inserted in the tubes. The temperature of the furnace was controlled to $\pm 10^{\circ}C$. Upon completion of the anneal the specimen was quenched by breaking the "Vycor" tube under water.

Replicas for Electron Microscopy

The microstructures were replicated by conventional techniques. The specimens were electropolished in a solution of phosphoric acid, ethylene glycol, and ethyl alcohol prior to replication. The replica was a 0.5% solution of "Formvar" in dioxane. For added strength when the replica was stripped from the specimen, the replica was backed with a film that was formed from a solution of 2.0% "Parlodion" in amyl acetate. The replicas were dry stripped with "Scotch" tape and then shadowed with uranium at an angle of 45°. The "Parlodion" backing was dissolved in amyl acetate before the replica was observed in the electron microscope.

IDENTIFICATION OF PHASES

Al-Ni Diffusion Couples

Metallographic examination of diffusion couples of aluminum and nickel bonded for five minutes at 565°C revealed two zones, as shown in Figure 1a. With an additional diffusion anneal of 48 hours at 600°C, the appearance of these two zones changed, and an additional zone appeared as shown in Figure 1b. The dark grey zone grew at the expense of the aluminum-rich zone until the latter was no longer visible. This effect has also been observed by Storcheim[5]. A new zone, richer in nickel, was also observed. The two aluminum-rich zones shown in Figure 1a were similar in appearance to those found in the step-pressed fuel element shown in Figure 2.

Samples of the aluminum-nickel zones were obtained for X-ray diffraction by a combination of mechanical and chemical removal of the aluminum. The aluminum was ground parallel to the interface until only a thin layer remained. This remaining aluminum was then dissolved electrolytically in a solution of citric acid, sodium chloride, and disodium phosphate as described by Fink and Willey[13] for isolating particles of Al_3Ni. The sample for X-ray diffraction was scraped from the exposed surface. The resulting pattern was attributed to a mixture of Al_3Ni and Al_3Ni_2. These results confirm the identification made by Storcheim[5]. From consideration of the phase diagram shown in Figure 3, the third zone observed after the 48-hour anneal is believed to be AlNi.

U-Ni Diffusion Couples

Diffusion couples of uranium and nickel bonded for five minutes at 565°C and annealed five days at 600°C were composed of three zones that were visible under bright-field illumination as shown in Figure 4a. When examined under polarized light, these zones were further resolved into a total of six zones. Three of the zones were anisotropic to polarized light, as shown in Figure 4b. The structure of these zones under the electron microscope is shown in Figure 5.

When additional specimens were annealed for ten days at 600°C, the couples fractured. Metallographic examination revealed that the zone of fracture was at the interface between the nickel and the first intermetallic compound. The surface of the uranium bearing the diffusion zones was scraped to provide a sample for X-ray diffraction. The resulting pattern was identified as UNi_5, with UO_2 also present.

When the couples were annealed for ten days at 700°C, seven zones were formed, as shown in Figure 6. The observation of the seventh zone confirmed the number of intermetallic phases predicted by the phase diagram given by Grogan and Pleasance[9]. The relative widths of the zones differed as a result of the variation of diffusion coefficients with temperature. When one of these couples was broken, the fracture occurred at the point indicated by the arrow at the left of Figure 6. The mating surfaces were sampled for X-ray diffraction. The zones adhering to the nickel were identified[9,10] as UNi_5, Compound Y, Compound X, and UNi_2, with a trace of UO_2 present. The zones adhering to the uranium were U_7Ni_9 and U_6Ni.

Another diffusion couple was inadvertently annealed at some temperature greater than 740°C because of a failure of the furnace controller. It is believed that the temperature was at least 740°C because metallographic examination revealed a eutectic structure which is indicative of melting, and 740°C is the lowest melting point in the uranium-nickel system. The microstructure resulting from this heat treatment is shown in Figure 7a. The work of Grogan and Pleasance showed that certain characteristic reactions occurred during the etching of uranium-nickel alloys in an equivolume mixture of nitric and acetic acids. The U_7Ni_9 phase was stained yellow, and the interface between this compound and U_5Ni_7 was delineated. When a specimen similar to that shown in Figure 7a was etched in this reagent, the predicted result was found to occur. Figure 7b shows the specimen after etching.

A selective etch was developed to aid in the identification of the zones. A 25% solution by volume of nitric acid was used as the electrolyte with the specimen as the anode, and a potential of six volts was applied. When the specimens shown in Figures 4 through 6 were etched in this reagent, the four nickel-rich zones were not attacked. When the specimen shown in Figure 7 was etched, the grey phase and the eutectic were severely attacked, leaving the white phase in relief. The white phase was removed mechanically and was identified as UNi_2 by X-ray diffraction. An additional specimen was scraped from the grey phase and the eutectic and was identified as a mixture of U_6Ni and U_7Ni_9.

From the above X-ray analyses, the relative attack of etchants, and a knowledge of the phase diagram, it was possible to identify the seven diffusion zones. The number of zones found in the couples confirms the number of intermetallic compounds indicated by Grogan and Pleasance in their phase diagram, which identified seven compounds as shown in Figure 8. The phase diagram that was published in the United States[8] identifies only four compounds.

As-Bonded Elements

The oxidation that occurred at the periphery of the uranium portion of the couple during the initial bonding served to identify the second phase found in the nickel-rich zone in as-bonded elements. After the anneal of five days at 600°C the oxide line appeared as shown in Figure 9a. The oxide line was broken by the diffusion and was dispersed throughout the three nickel-rich zones. The dispersed form of the oxide was very similar to the second phase found in as-bonded elements, as shown by the electron micrographs in Figures 9b and 9c. This fact, coupled with the X-ray identification of UO_2 with UNi_5 in the diffusion couples annealed 10 days at 600°C and 700°C, suggests that the second phase is UO_2. However, the possibility cannot be neglected that some combination with nickel had occurred, and that the second phase was a uranium-nickel-oxygen compound.

To aid in the identification of the zones observed in as-bonded elements, plate specimens were annealed further to study the growth of existing zones, the location of newly formed zones, and the effect of such treatment on the dispersed UO_2 particles observed in the nickel-rich zone. A special element having a nickel thickness of 0.0015 inch was used to prevent the formation of uranium-aluminum compounds as a result of depletion of the nickel during annealing.

The changes in the microstructure resulting from diffusion anneals at 600°C for 10, 30, 90, and 120 minutes are illustrated by the electron micrographs in Figure 10. Three important changes from the as-pressed structure shown in Figure 9c were observed: First, new uranium-nickel zones were formed at the interface between the two original zones. Second, the UO_2 particles coalesced into a zone frequently seen in specimens from as-bonded elements but not observed in uranium-nickel diffusion couples. The fact that, upon additional annealing, the new zone appeared and the UO_2 particles disappeared has been taken as evidence for the coalescence of the particles. It is also possible that the UO_2 particles had dissolved and subsequently combined with nickel to form a ternary layer. Third, the zone formed by the coalescence of the UO_2 particles disappeared during the 120-minute anneals, probably by dissolution into the uranium.

With longer anneals of 24 hours at 600°C the changes in the microstructure were visible with the light microscope, as shown in Figure 11. Two changes were observed in the aluminum-nickel diffusion zones. The first was the growth of the Al_3Ni_2 zone at the expense of the Al_3Ni to the extent that the latter was no longer visible. The second change was the appearance of a third zone which was similar to the zone found in the diffusion couple shown in Figure 1b and believed to be AlNi.

As shown in Figure 11a, the two original uranium-nickel zones grew, and two new zones became visible. The zones were similar to those seen in the diffusion couples. When the specimen was viewed under polarized light, grains were visible in the uranium-rich zone as shown in Figure 11b. Since the U_6Ni layer identified on the diffusion couples described previously was anisotropic, it is believed that this zone is also U_6Ni.

The above study of the changes in microstructure of the bond with additional annealing made it possible to identify the zones in as-bonded elements. The fact that the new zones were observed to form at the interface between the two original zones has been taken as evidence that the two original zones are the extreme intermetallic compounds identified by the phase diagram, U_6Ni and UNi_5. The new zones were identified by correlation with the relative widths of the zones identified in the couples annealed at 600°C.

RELATIONSHIP BETWEEN MICROSTRUCTURE AND PROPERTIES

During the course of development of the step-pressing process for plate elements, it was found that increased bond strength was achieved by etching the core in nitric acid at 30°C rather than at 50°C prior to electroplating. This increase in strength is illustrated by the histograms of bond strength shown in Figure 12a. At the time this work was done it had been thought that the increased bond strength was due in part to the increased surface roughness that resulted from the etch at the lower temperature, and also to the minimization of oxidation of the core during transfer from the etch to the electroplating bath.

Examination of specimens fabricated by these two procedures revealed distinct differences in the microstructures. The bond on an element etched in acid at 50°C had three zones between the nickel and the uranium, U_6Ni, the oxide layer, and UNi_5, whereas the bond on the element that was etched at 30°C had only the two uranium-nickel intermetallics. This difference is illustrated in Figure 12b.

Routine bond strength tests on tubular fuel elements established a significant difference in strength between the inside and outside bonds, regardless of fabricating conditions, as shown in Figure 13a. Examination of the two bonds with the electron microscope revealed a difference in structure similar to that observed in plate elements. The inside bond had the U_6Ni, oxide, and UNi_5 layers present, whereas the outside bond had only the two uranium-nickel intermetallics, as shown in Figure 13b. Thus, as in the case of the plate elements, the occurrence of the oxide layer correlated with the decreased bond strength.

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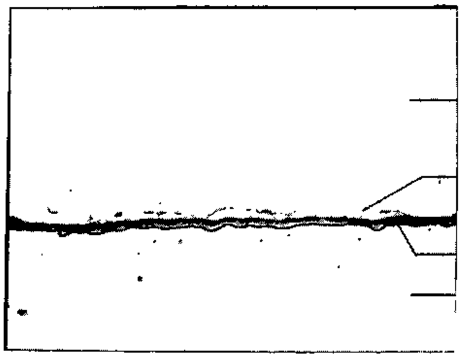
FIGURE 1



AS POLISHED **MAG. 500 X**

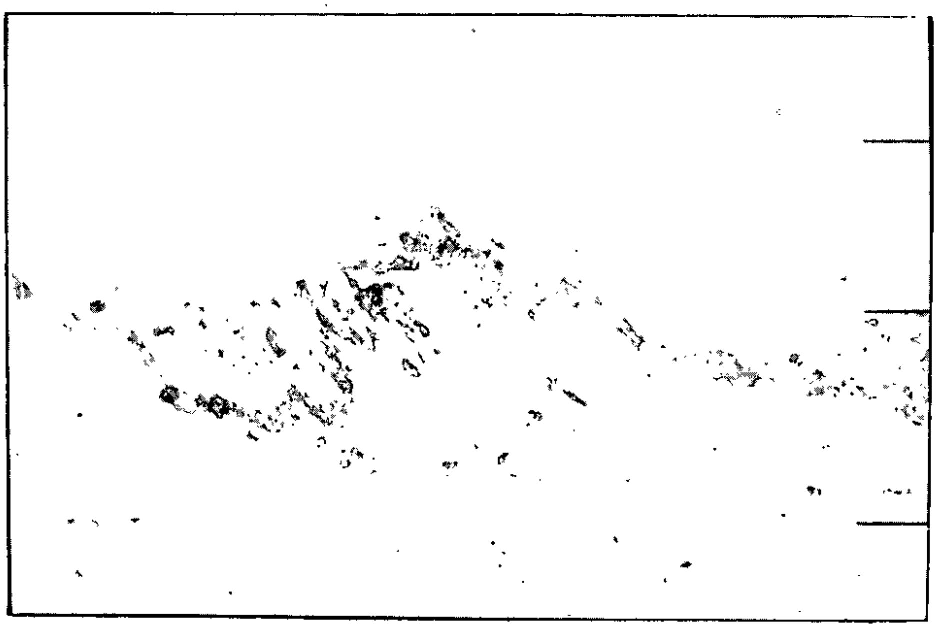
1a. Diffusion zones formed in an aluminum-nickel couple after bonding at 8000 psi and 565°C for 5 minutes.

AS POLISHED **MAG. 445 X**

1b. Diffusion zones formed in an aluminum-nickel couple that was bonded as described at left and annealed for 48 hours at 600°C.

DIFFUSION ZONES IN ALUMINUM - NICKEL COUPLES

FIGURE 2

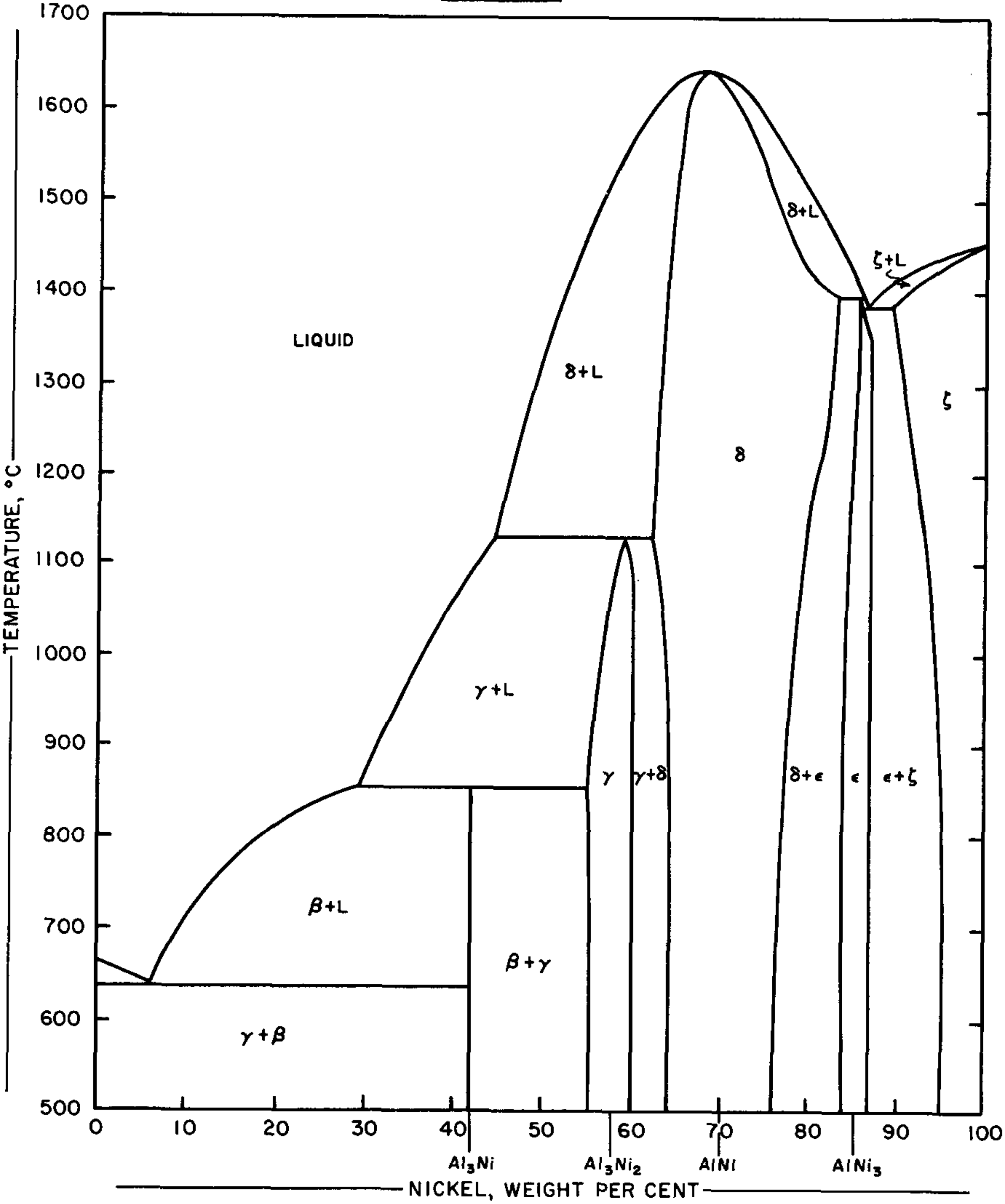


ELECTROPOLISHED **MAG. 415 X**

Microstructure of an as-bonded fuel element showing in particular the two zones formed between the nickel and the aluminum. Note the similarity of these zones with those formed in the diffusion couples.

MICROSTRUCTURE OF THE BOND IN A STEP-PRESSED FUEL ELEMENT

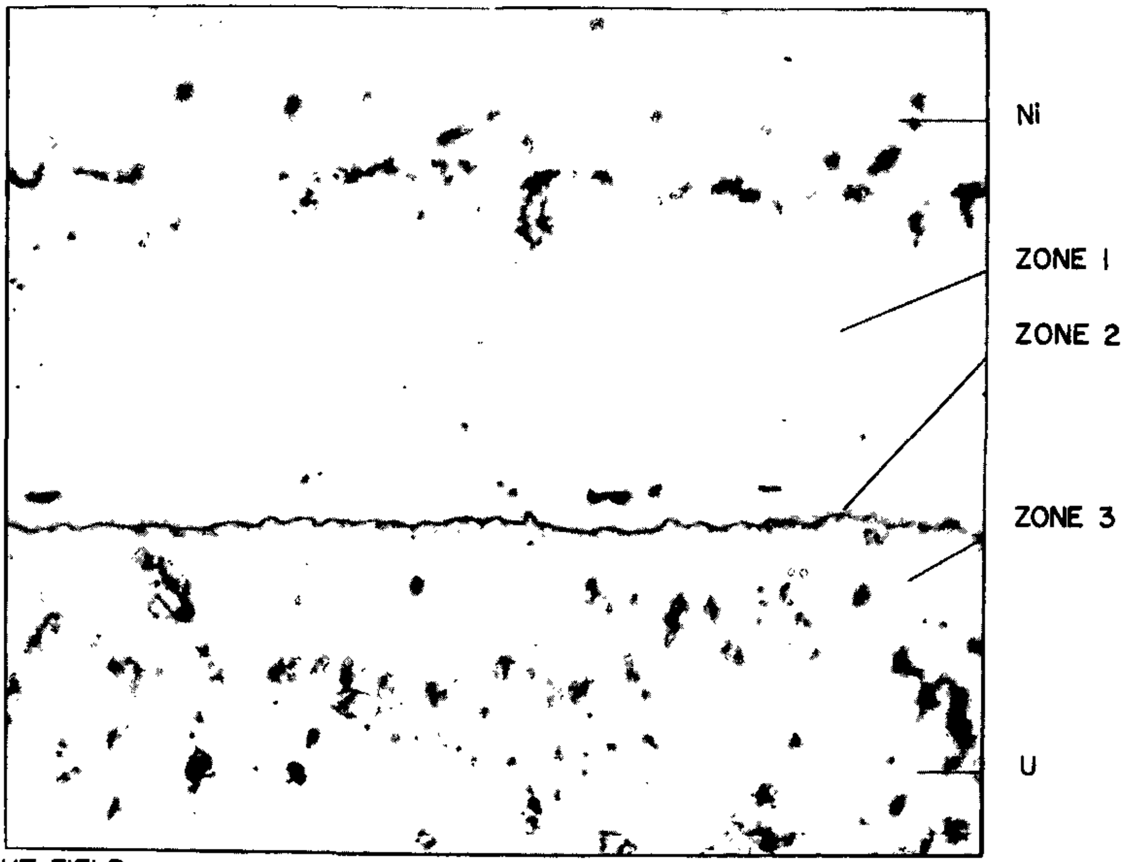
FIGURE 3



ALUMINUM-NICKEL PHASE DIAGRAM

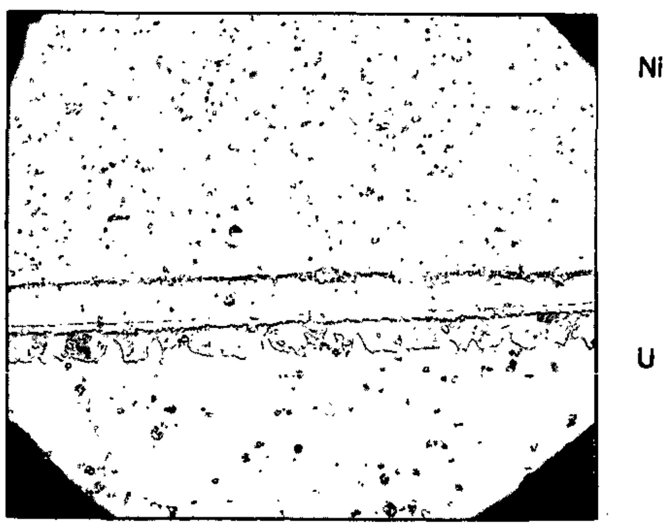
FINK AND WILLEY

FIGURE 4



BRIGHT FIELD MAG. 1100 X

4a. Note that three zones are visible.

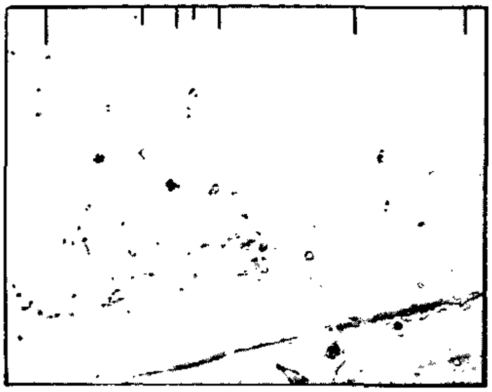


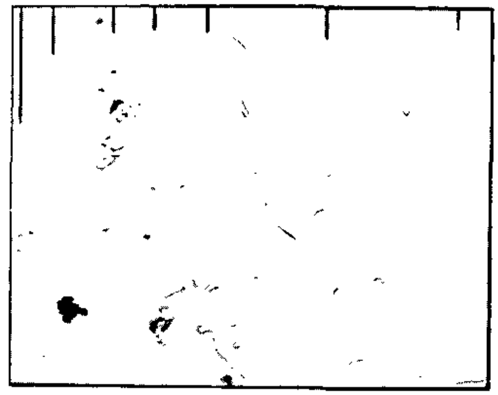
POLARIZED LIGHT MAG. 213 X

4b. Zone 1 in Figure 4a is now resolvable into three zones. Zone 2 is resolvable into two zones.

DIFFUSION ZONES IN A URANIUM - NICKEL COUPLE ANNEALED FIVE DAYS AT 600°C

FIGURE 5





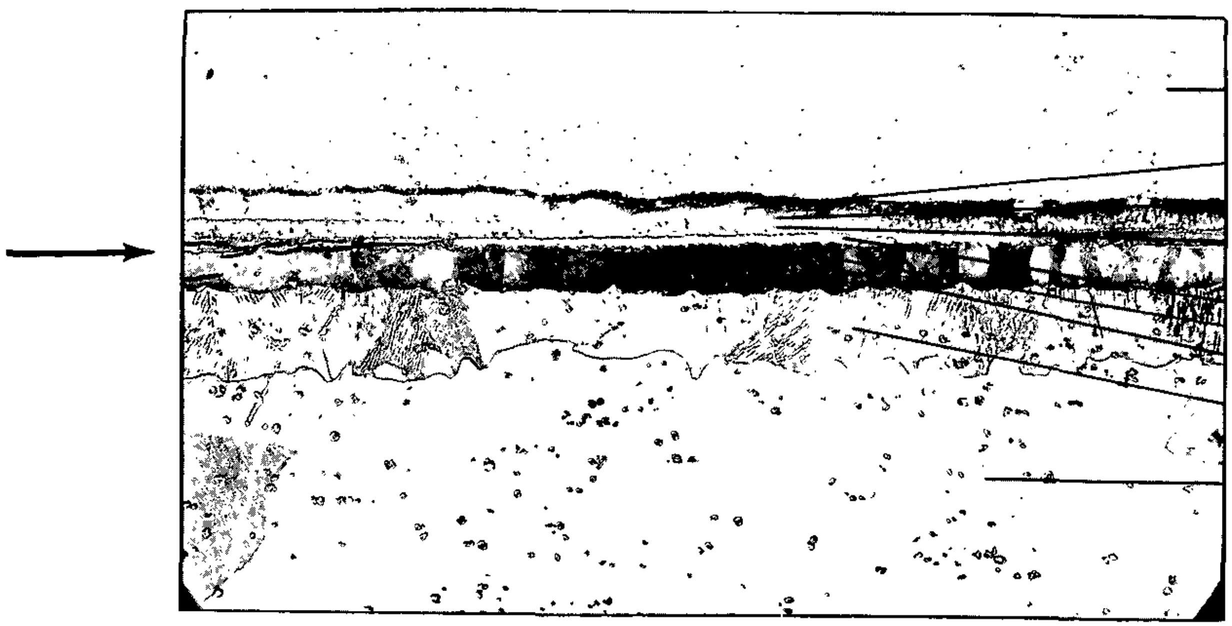
MAG. 1650 X

MAG. 3696 X

Electron micrographs of the uranium-nickel intermetallics formed by diffusion at 600°C for five days.

ELECTRON MICROGRAPH OF URANIUM - NICKEL DIFFUSION ZONES

FIGURE 6



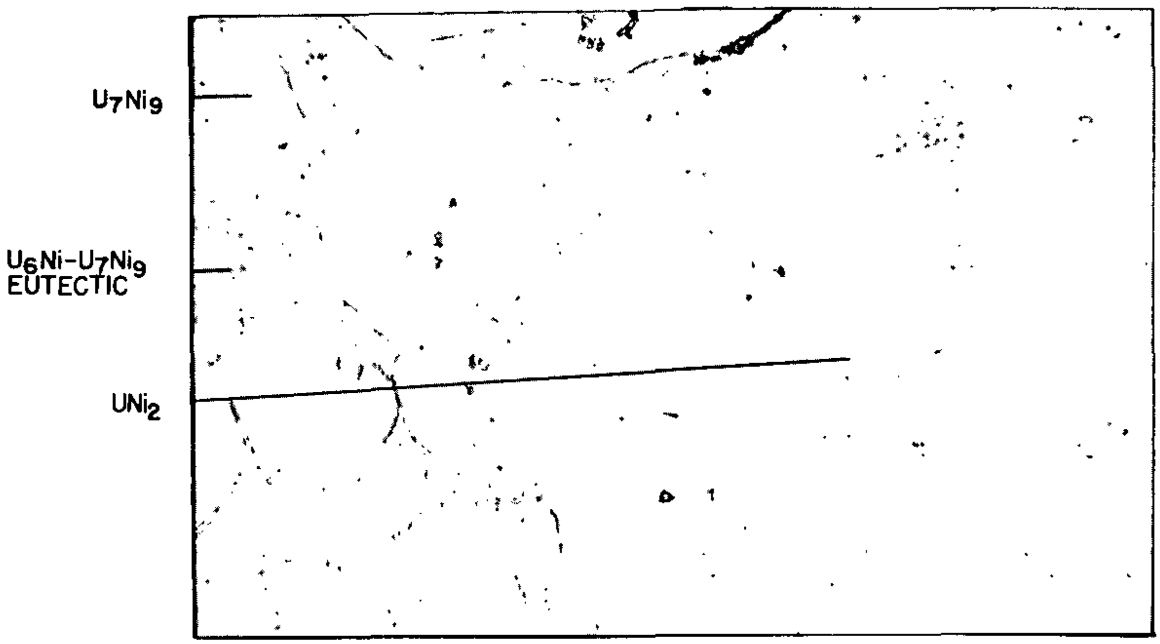
POLARIZED LIGHT

MAG. 220 X

When the couple was broken, the fracture occurred along the line indicated by the arrow. The zones adhering to the surfaces were then identified by X-ray diffraction.

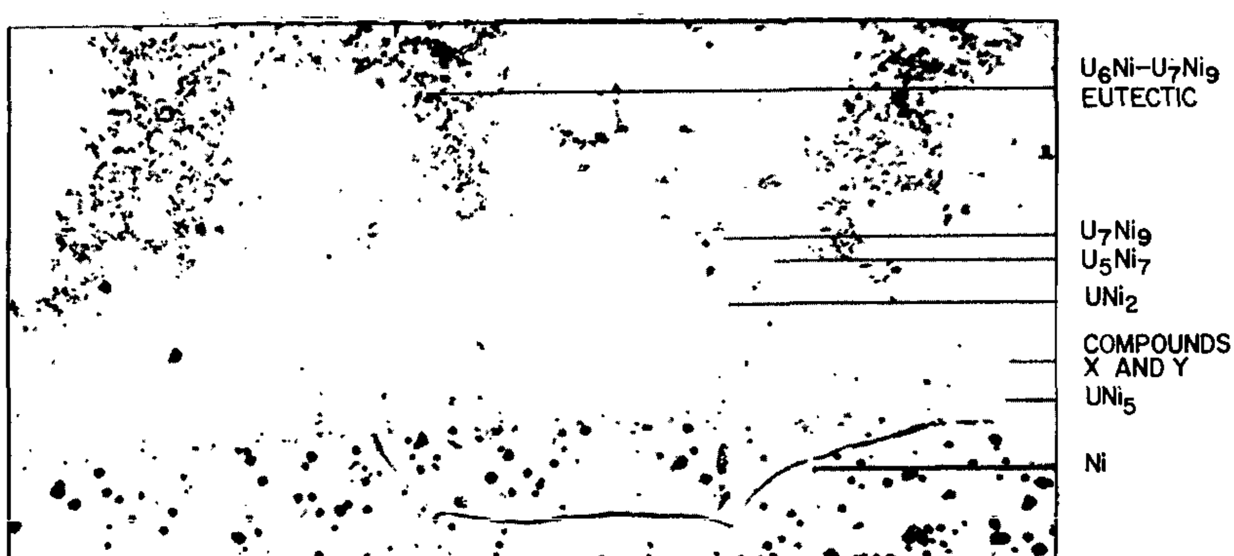
DIFFUSION ZONES IN A URANIUM -NICKEL COUPLE ANNEALED TEN DAYS AT 700°C

FIGURE 7



H_3PO_4 ELECTROPOLISHED MAG. 182 X

7a. Uranium-nickel alloy couple. The structure shown was found throughout the sample.

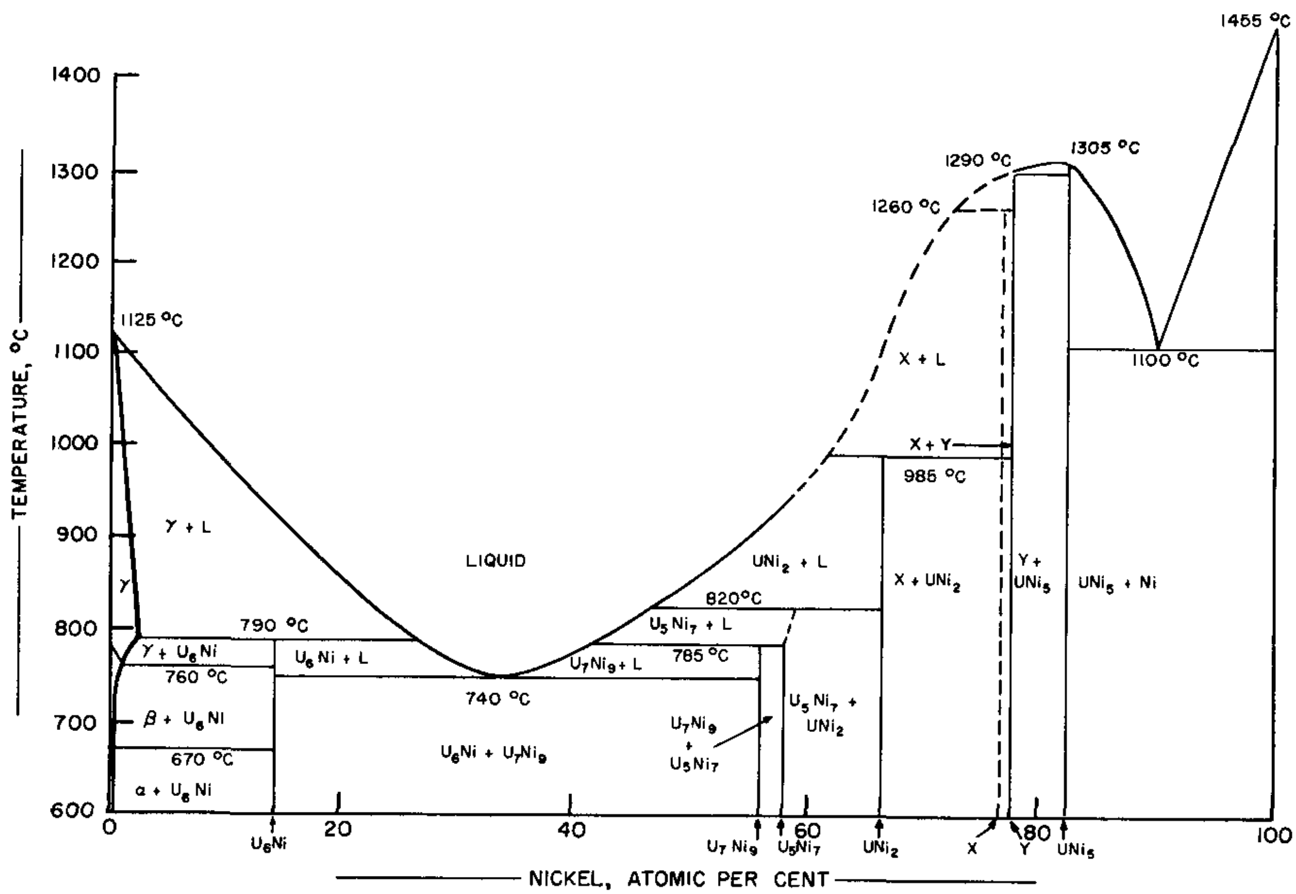


HNO_3-$HC_2H_3O_2$ ETCH MAG. 163 X

7b. Uranium-nickel alloy couple. The etch stained the U_7Ni_9 and delineated the U_5Ni_7 layer.

MICROSTRUCTURE OF A URANIUM – NICKEL ALLOY

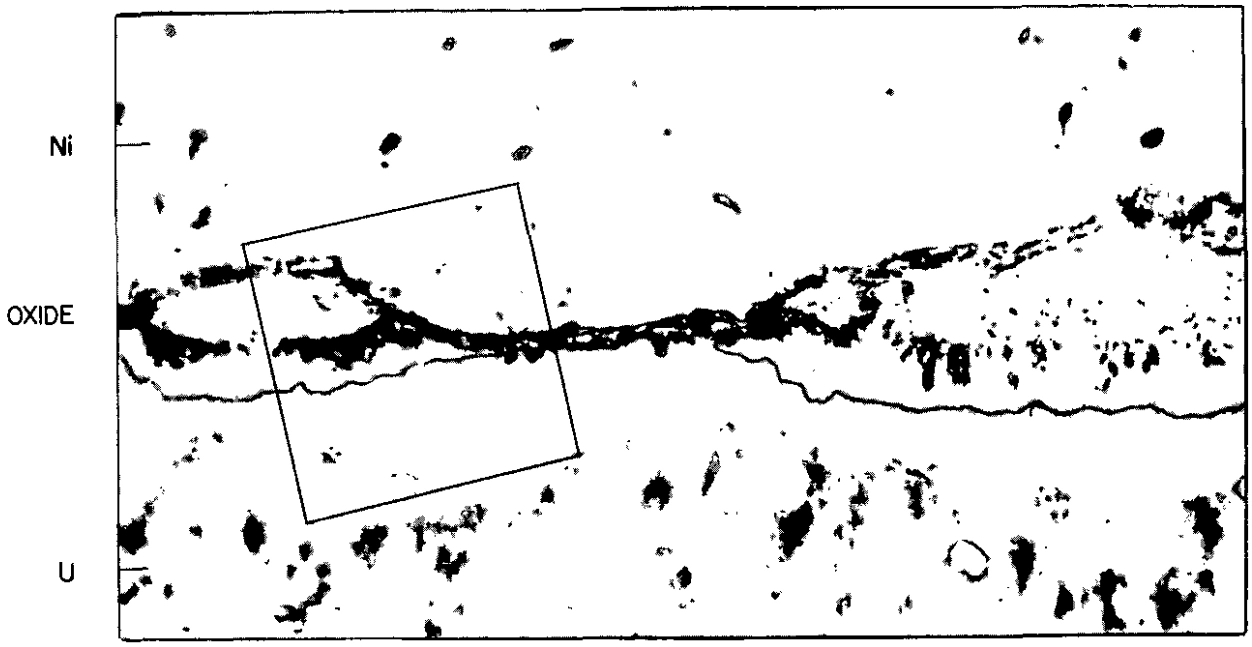
FIGURE 8



URANIUM-NICKEL PHASE DIAGRAM

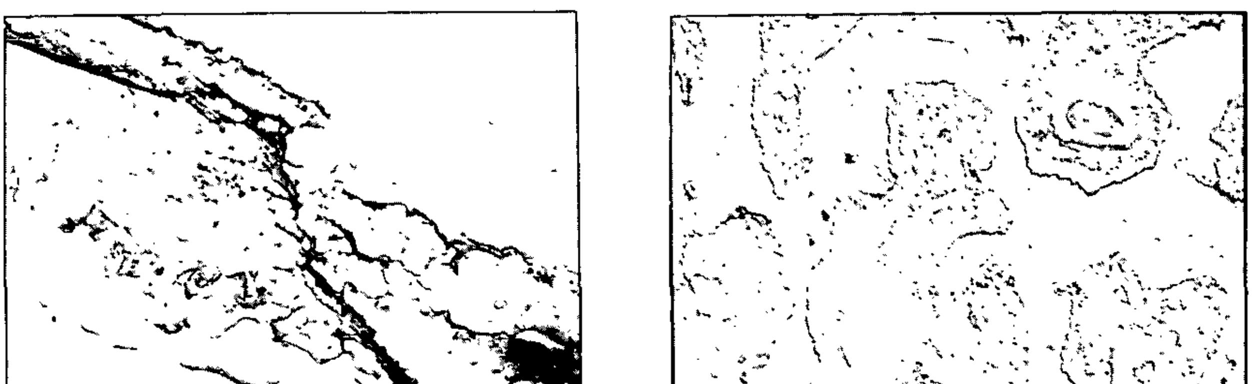
GROGAN AND PLEASANCE

FIGURE 9



ELECTROPOLISHED MAG. 854 X

9a. Light micrograph of the edge of U-Ni couple annealed five days at 600°C. The line of oxide is shown at the left. Note the break-up on the right where diffusion has occurred.



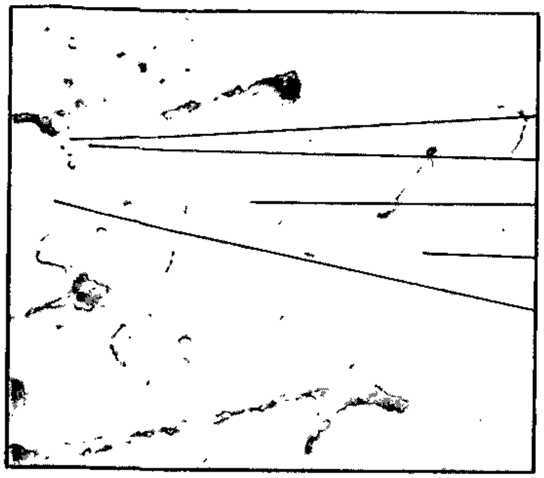
DIFFUSION COUPLE MAG. 2500 X

PLATE FUEL ELEMENT MAG. 5600 X

9b and 9c. Figure 9b shows an electron micrograph of the outlined area in Figure 9a. Note the similarity between the broken oxide particles in this couple and the second phase observed in the nickel-rich zone of the as-bonded element shown in Figure 9c.

EFFECT OF UO_2 ON THE MICROSTRUCTURE OF A URANIUM-NICKEL DIFFUSION COUPLE

FIGURE 10



MAG. 8667 X

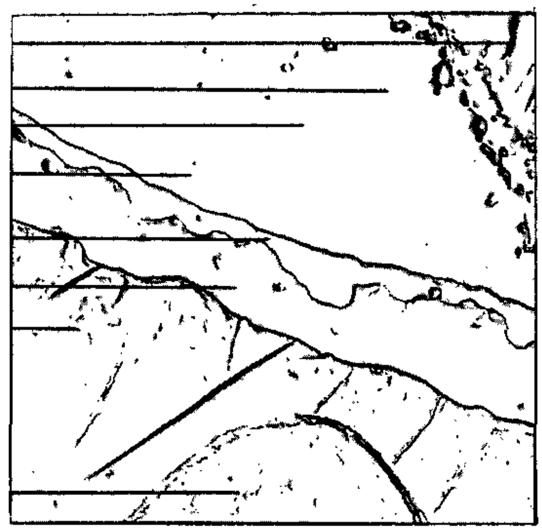
10a. Specimen annealed 10 minutes at 600°C. A new intermetallic zone was visible, but no change in the oxide particles was seen.

MAG. 9577 X

10b. Specimen annealed 30 minutes at 600°C. The coalescence of the UO_2 into a continuous zone was complete. Two new zones were visible.

MAG. 9577 X

10c. Specimen annealed 90 minutes at 600°C. Three new zones were visible. The oxide layer grew.

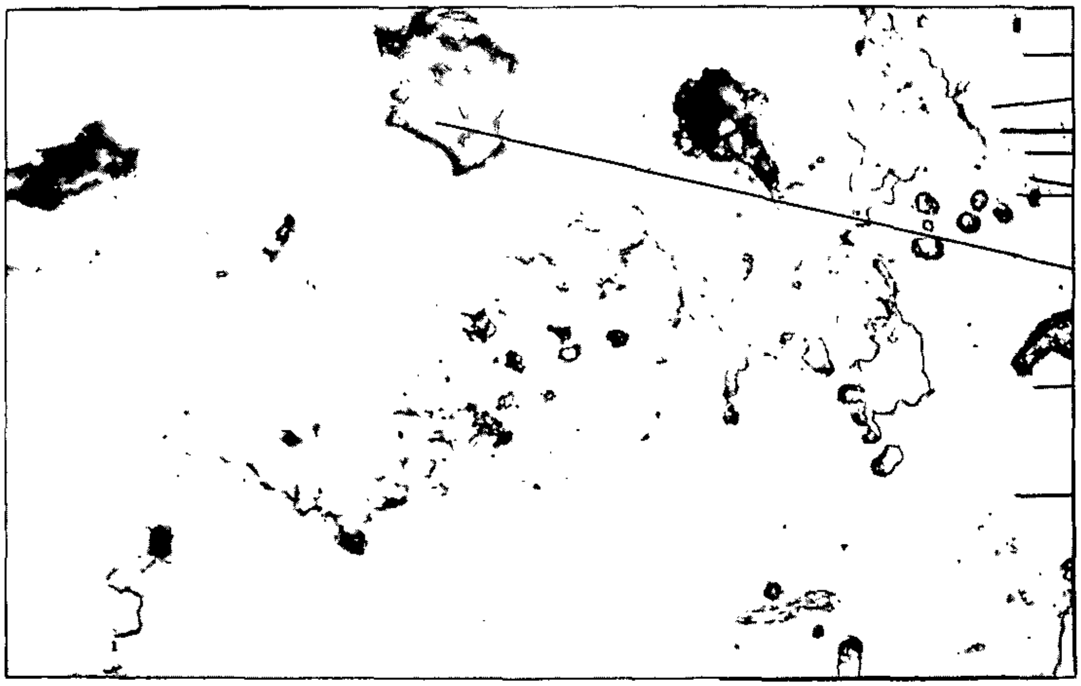


MAG. 9791 X

10d. Specimen annealed 120 minutes at 600°C. The continuous UO_2 zone had disappeared. Six diffusion zones were visible.

ELECTRON MICROGRAPHS OF THE BOND AFTER BRIEF ANNEALING

FIGURE 11



ELECTROPOLISHED MAG. 500 X

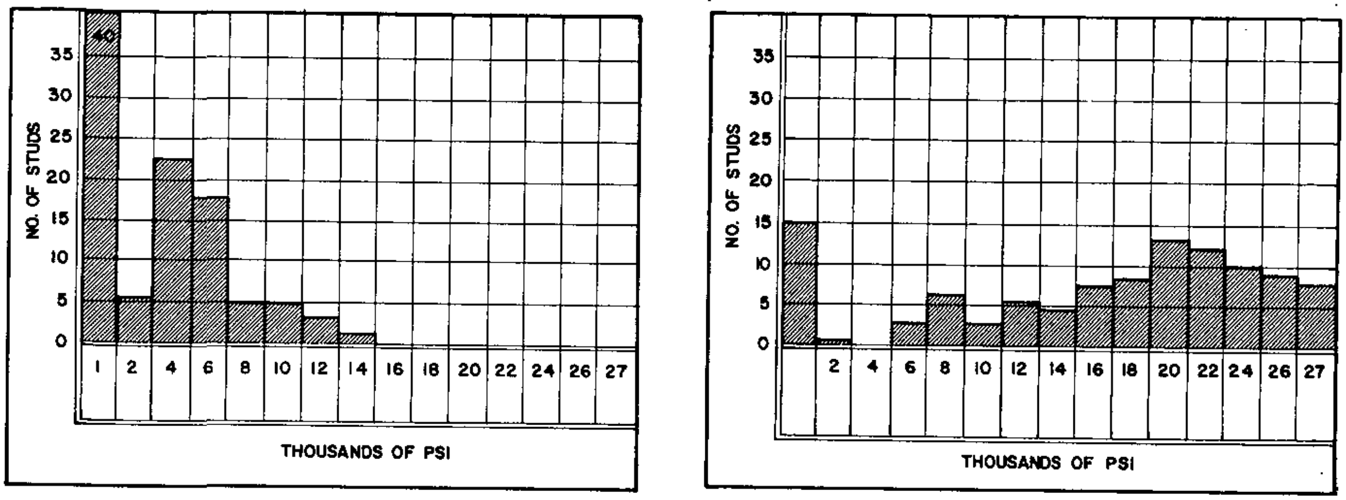
11a. Microstructure of an as-bonded element after an additional diffusion anneal of 24 hours at 600°C. The Al_3Ni_2 zone has grown at the expense of the Al_3Ni, and a new zone believed to be AlNi has formed. Two new U-Ni zones are visible.

POLARIZED LIGHT MAG. 500X

11b. Same specimen as shown above. An island of the uranium-rich compound in a matrix of uranium is shown. Note the grains visible in this compound.

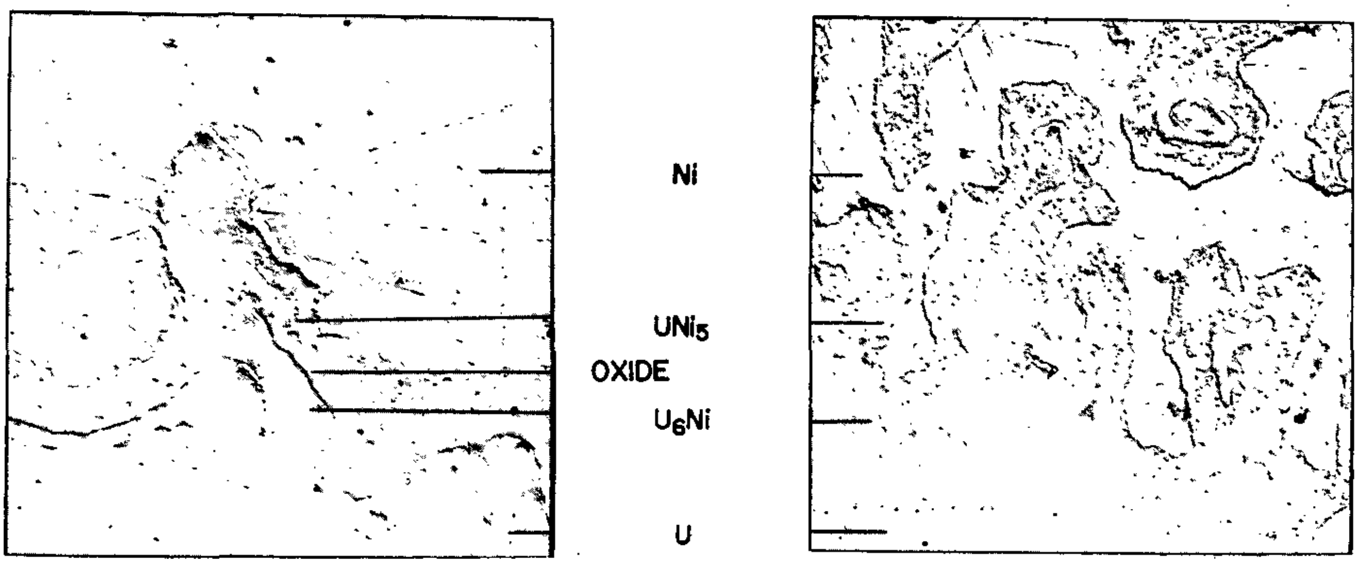
MICROSTRUCTURE OF THE BOND AFTER EXTENDED ANNEALING

FIGURE 12



50°C ETCH 30°C ETCH

12a. Histograms of bond strengths of elements etched in 50°C acid and in 30°C acid.

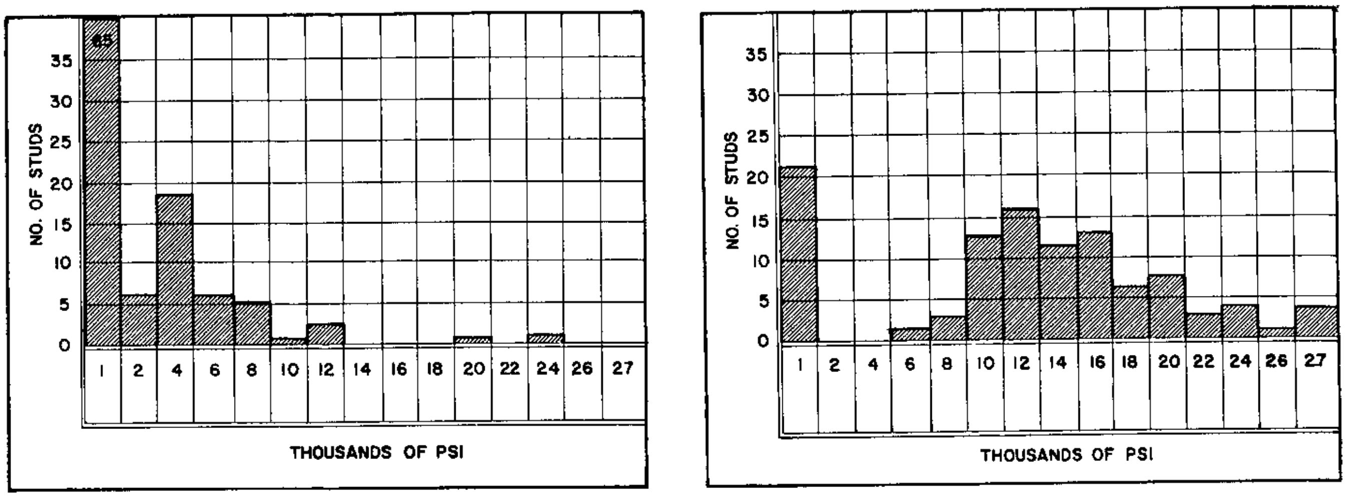


50°C ETCH MAG. 6981 X 30°C ETCH MAG. 5264 X

12b. Microstructures of elements etched in 50°C acid and in 30°C acid. Note the presence of the oxide layer in the element etched at 50°C but not in the element etched at 30°C.

EFFECT OF FABRICATING CONDITIONS ON THE BOND STRENGTH AND MICROSTRUCTURE OF PLATE FUEL ELEMENTS

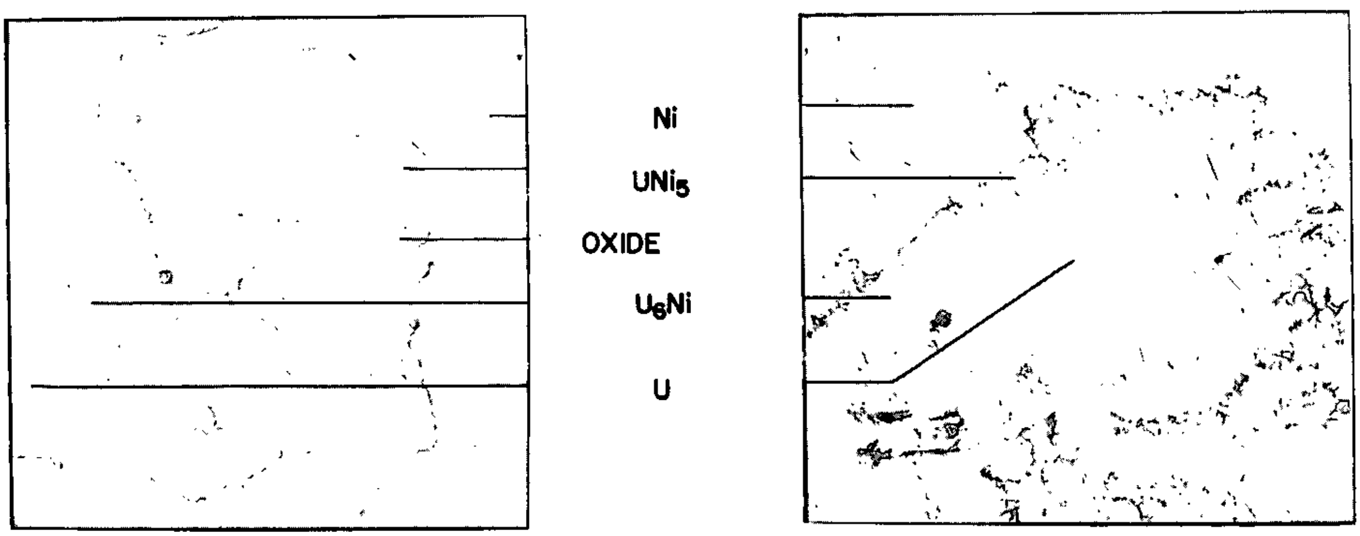
FIGURE 13



INSIDE BOND

OUTSIDE BOND

13a. Histograms of bond strengths in tubular fuel elements.



INSIDE BOND MAG. 5600 X

OUTSIDE BOND MAG. 3700 X

13b. Microstructure of bonds in tubular elements. Note the presence of the oxide layer in the inside bond but not in the outside bond.

STRENGTH AND MICROSTRUCTURE OF THE BONDS IN TUBULAR FUEL ELEMENTS

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March 27, 1958

JC Andrews
cc: JWM

Dr. M. H. Wahl, Assistant Director
Technical Division - AED
Savannah River Laboratory
E. I. du Pont de Nemours and Company
Aiken, South Carolina

Attention: Dr. J. W. Morris

Dear Dr. Wahl:

We have reviewed the following classified document as to its suitability for release under the Civilian Application Program and agree that it may be released as written:

"DP-248 by C. L. Angerman"

Your office has been notified of this action by telephone.

Very truly yours,

J. V. Levy

J. V. Levy, Director
Industrial & Technical
Services Division

see letter
dated 5/21/58

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ATOMIC ENERGY COMMISSION
SAVANNAH RIVER OPERATIONS OFFICE
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May 21, 1958

Mr. Hood Worthington, Director
Technical Division - AED
Explosives Department
E. I. du Pont de Nemours & Company
Wilmington 98, Delaware

Dear Mr. Worthington:

By our letter of March 27, 1958, we released the following classified document under the Civilian Application Program:

DP-248 -- "Electron Metallography of the Al-Ni-U Bond", by C. L. Angerman

By memo of May 7, 1958, from J. H. Kahn, Assistant Chief, Declassification Branch, Division of Classification, USAEC, we have been advised that this document has been declassified without deletions.

Patent review has been made and there is no objection to release on an unclassified basis.

Please notify all holders of this document of this action, citing the above authorization.

Very truly yours,

J. V. Levy, Director
Industrial & Technical Services Division

CC: Dr. M. H. Wahl (2) - Attn: Dr. J. W. Morris
F. A. Robertson, Patent Branch, SROO

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BCC: Hood Worthington -
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~~J. W. Morris~~ -
J. ~~C. Andrews~~

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INCORPORATED
Wilmington, Delaware

EXPLOSIVES DEPARTMENT

March 31, 1958

Mr. J. H. Kruth, Chief (2)
Classification Branch
Industrial & Technical Services Division
United States Atomic Energy Commission
Aiken, South Carolina

Dear Mr. Kruth:

CLASSIFICATION CONSIDERATIONS - DP-248

The above report, "Electron Metallography of the Al-Ni-U Bond, by C. L. Angerman, has been submitted to your office and reviewed from the point of view of classification.

It is recommended that this report be submitted for declassification under topics 1-250 and 1-251 of OC DOC-44. The basic information of this report is scientific information of general value in fuel element fabrication and the references to cladding methods under development at Savannah River are of such a general nature that no essential technology is disclosed. The fuel element shapes referred to are of course unclassified by topic 310.1.1 of the SRO Classification Guide.

Very truly yours,

S. A. McNeight
Technical Assistant

SAMcN:ESC

DP- 248 - 2nd.

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J. C. Andrews - TIS File
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SAVANNAH RIVER LABORATORY

January 22, 1958

Mr. Joel V. Levy, Director (2)
Industrial and Technical Services Division
Savannah River Operations Office
U. S. Atomic Energy Commission
Post Office Box A
Aiken, South Carolina

Dear Mr. Levy:

PROPOSED PUBLICATION - DP-248

Attached for review for suitability for release under the Civilian Application Program are five copies of the following classified report:

Electron Metallography of the Al-Ni-U Bond
by C. L. Angerman

Please contact my office if you have any comments. The report will be released when approval is received, but not until after 14 days from the date shown above.

Yours very truly,

ORIGINAL SIGNED BY
M. H. WAHL

M. H. Wahl, Asst. Director
Technical Division, AED

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U. S. Atomic Energy Commission

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Author C. L. Angerman Al-Ni-U

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Category Metallurgy & Ceramics Approved by [signature]

Abstract check with SWO Approved by [signature]

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