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INTERIM EVALUATION OF NICKEL PLATE ON
ALUMINUM-JACKETED FUEL ELEMENTS

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INTERIM EVALUATION OF NICKEL PLATED

ALUMINUM-JACKETED FUEL ELEMENTS

G. F. Jacky

February 8, 1960

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INTERIUM EVALUATION OF NICKEL PLATE ON ALUMINUM-JACKETED FUEL ELEMENTS

INTRODUCTION

Nickel plating on the coolant contacting surfaces of aluminum-jacketed fuel elements is highly attractive for increasing failure-resistance capabilities. Potential benefits include a highly corrosion-resistant coating for severe localized conditions, reduction of mechanical damage to fuel element jackets, improved fuel element alignment (by reducing friction between fuel elements and process tube ribs) and probably lower overall surface temperatures due to reduction in corrosion product film. With improved corrosion resistance, neutron economy might also be realized. For example, substitution of a 0.5-mil thick nickel plate for 15-mils thickness of aluminum jacket would result in no reactivity loss and permit a concomitant increase in uranium volume, or in coolant flow annulus.

Attendant problems include providing an adherent continuous plate of uniform thickness and possibly contamination of reactor effluent by radio-nickel, cobalt, and phosphorous. To determine gravity of the contamination problem, an irradiation test¹ was carried out in early 1959. The increase in reactor effluent contamination was insignificant,² the validity of this conclusion being limited only by the size of the test and the exposure (four charges to ~400 MWD/T). Another highly encouraging result was that 1/2-mil thick chemical-nickel plate prevented corrosion of underlying C-64 alloy aluminum in a severe "hot-spot", with an estimated surface temperature of 325-350° C for over 100 hours.³ On the debit side, it was found that gross sloughing of the nickel plate had occurred. Development and testing work was carried out to determine the cause and a solution to the adherence problem. Studies were limited to the behavior of chemically-deposited nickel because of the unique capability of the process to deposit a coating of uniform thickness in the 0.1 - 0.2 mils thick range, regardless of the geometry of the plated piece. Based on ex-reactor tests, a readily applicable method for significantly improving plate adherence has been developed, as summarized below.

1. HW-58179 A, "Production Test IP-207-A-3 FP, Evaluation of Effluent Contamination Problems Associated with Nickel-Coated Fuel Elements", R. E. Hall and R. B. Hall, November 14, 1958.
2. HW-58179 C, "Irradiation Summary Report - PT-IP-207-A-FP", R. B. Hall and R. E. Hall.
3. HW-62441, "Post Irradiation Examination of a Nickel Plated Fuel Element from PT-IP-207-A (RM 306)", W. J. Gruber, October 30, 1960.

The writer wishes to acknowledge the assistance of D. W. Shannon and R. Sorenson for their work in developing an adherence test, and P. L. Lee who carried out much of the adherence testing and evaluation.

SUMMARY

Examination of irradiated nickel-plated fuel element and ex-reactor tests did not indicate that nickel sloughing was due to corrosion of the underlying aluminum or to generation of cracks in the nickel plate as a result of thermal cycling. The severest in-reactor sloughing occurred in regions of high neutron flux, but upstream of the maximum flux, and was significantly reduced in regions of maximum surface temperature. Metallographic sections of irradiated fuel elements having slightly separated nickel plate and aluminum substrate did not reveal corrosion undercutting of the nickel. Efforts to reproduce extensive nickel sloughing by ex-reactor thermal cycling and by long-term exposure to 120 and 165° C reactor process water were unsuccessful.

A third possible cause of sloughing suggested by W. E. Tragert (GERL) is absorption of atomic hydrogen generated by radiolysis of water. Thus, molecular hydrogen may collect at even sub-microscopic voids within the nickel or at the nickel-aluminum interface, and the accumulated pressure may be sufficient to cause blistering and sloughing of the plate. The validity of this mechanism was checked experimentally employing electrolytically generated hydrogen. It was found that the nickel plate could be blistered to the extent that it essentially completely separated from the aluminum, and the blisters resembled those found on irradiated fuel elements. An adherence test was developed, employing electrolytically generated hydrogen.

Adherence of chemically deposited nickel and aluminum is vastly improved by post-plating heat treatment as revealed by both conventional bend testing* and by the electrolytic-hydrogen test. Merely autoclaving for 20 hours at 165-170° C produces a measurable improvement. Major additional improvement is achieved by baking one hour and longer at 300-400° C. Comparatively, in the electrolytic hydrogen blistering test, autoclaved unbaked samples averaged about 40 per cent of surface area blistered, with values as high as 100 per cent; samples of the same material, baked for three hours at 300° C did not blister. Consistent improvement is realized only if the plate has good adherence in the as-plated condition, as determined by bend testing.

Baking at times and temperatures which produce a significant improvement in aluminum-nickel adherence causes no measurable deterioration in Al-Si - U bond quality as revealed by tensile strength and ultrasonic testing and by metallographic examination. There is some indication that baking toughens the bond due to formation of additional UAl₃, one of the more ductile compounds formed during canning.

* Poorly adherent plate will separate from the base metal at fractures produced by repeated bending.

Considering future irradiation testing, possible hazards to the continuity of reactor operation were further assessed. These include coolant flow restriction from sloughed nickel, and accelerated corrosion of aluminum at discontinuities in the nickel plate. A flow test in a BDF-type process tube, using 2 - 3-mils thick chemical-nickel plate, indicated that a flow restriction from sloughed chemical plate is highly unlikely; results are particularly significant, considering that the plate for in-reactor service will probably be 0.5 mils or less in thickness. Corrosion testing chemically plated C-64 alloy aluminum in reactor process water flow tubes revealed no accelerated attack of aluminum at discontinuities in the plating after eight weeks at 65° C, 22 weeks at 120° C, and 18 weeks at 160° C. Likewise, examination of irradiated fuel elements did not reveal evidence of galvanic corrosion. Exploratory testing of nickel-plated C-64 alloy aluminum in 360° C demineralized water autoclaves indicated the potential of nickel plate for minimizing or eliminating aluminum corrosion, even with discontinuities up to 1/16 inches diameter in the plating.

CONCLUSIONS

It appears desirable to resume in-reactor testing, considering the demonstrated potential of chemically-deposited nickel plate to protect aluminum from "hot-spot" corrosion, the encouraging results of the reactor effluent contamination test, and the improvement in plate adherence achieved by baking. A primary objective should be to determine if plate adherence is adequate. Secondly, additional effluent contamination data and experience on corrosion behavior is desirable.

DISCUSSION

PT-IP-207-A Summary

Production test IP-207-A⁴ was carried out primarily to determine if a nickel plate on the coolant contacting surfaces of fuel elements would cause intolerable contamination of reactor effluent. C-64 alloy jacket I & E fuel elements were used with both electro- and chemically-deposited nickel plating. The latter was of particular interest from effluent contamination standpoint since the plate is 6-8 wt. per cent phosphorous. Electroplating was done by the HLO-Fuel Element Fabrication Laboratory, and the chemical plating was done by an offsite vendor.

The test was charged in C Reactor, February 18, 1959, using the OCD facility with the reactor at full power. Exposure conditions may be summarized:

4. op. cit., HW-58179, Page 2.

<u>Tube No.</u>	<u>Material</u>	<u>Days In</u>	<u>Exposure MWD/T</u>	<u>Power Factor</u>
2072-C	1 mil electroplate	64	406	1.164
1469-C	1 mil electroplate	63	382	1.091
1468-C	1 mil chemplate	69	397	1.149
1675-C	0.5 mil chemplate	63	371	1.086
2572-C	Unplated	40*	265	1.205
2474-C	Unplated	96	598	1.094

* Discharged because of a tube leak

During the course of the irradiation, intermittent high Pannelit readings were noted on tube 2072-C and 1469-C (electroplated) and on April 25, C Reactor was shut down, possibly due to a high Pannelit on tube 2072-C. Examination of the fuel elements from the two electroplated charges and the single 1/2-mil thick chemical plate revealed that extensive sloughing of the nickel had taken place, and the remaining one-mil thick chemical plate charge was also discharged. Fairly conclusive reactor effluent activity data did not indicate a contamination problem,⁵ and examination of the irradiated fuel elements and other tests were carried out to determine the cause and remedy for sloughing of the nickel plate.

Examination of Irradiated Fuel Elements

Examination of irradiated fuel elements did not indicate that sloughing of the nickel plate was due to corrosion of the underlying aluminum or to generation of cracks in the nickel plate as a result of thermal cycling. There is substantial evidence that radiolytically generated atomic hydrogen may have diffused into the plate and caused blistering in poorly adherent areas.

Weight-loss data and underwater visual examination results are presented in Figure 1. Considering weight losses as well as appearance indicative of nickel sloughing, these data reveal the following:

1. The severest sloughing generally occurred in regions of high neutron flux, but upstream of the maximum flux region. This indicates a dependence on flux but not on mechanical strain of the fuel element (which would maximize with flux).
2. Sloughing was notably reduced in the regions having the highest surface temperature, indicating little or no effect of the aluminum corrosion rate, or of thermal cycling.

5. op. cit., HW-58179 C, Page 2.

3. In regions where extensive sloughing was observed and high weight losses occurred, behavior was generally erratic, indicating a difference in the quality of the plate.
4. Sloughing was reduced with increased nickel thickness, comparing the 1/2-mil and one-mil thick chemical plate, and considering that all of the observed electroplate sloughing occurred internally. (The bore of the electroplated elements was known to have thin spots, due to difficulty in plating.)

It should be added that the obvious relationship between adherence and plate thickness may be erroneous; the observed difference may be due to adhesion per se, since the two thicknesses of chemical plate were plated in different batches. Likewise, the difficulty in achieving a uniform thickness in the bore of the electroplated fuel elements, due to shielding by insulators on the internal anode, may have caused poor adherence.

Metallographic examination⁶ of an irradiated fuel element from having 1/2-mil thick chemical plate indicated the nickel plate probably prevented an in-reactor failure from corrosion in a "hot-spot" area, and supported evidence from under-water examination that sloughing was not due to corrosion or cracking of the plate from thermal cycling.

Salient results are:

1. The surface temperature in the "hot-spot" region was estimated at 325-350° C for a period of over 100 hours. The nickel plate was in good condition with no evidence of cracks, blisters, or sloughing.
2. At the fringe areas of the "hot-spot", the aluminum and nickel had separated, and intergranular attack of the aluminum occurred. Absence of aluminum corrosion product between separated nickel and aluminum and the matching contour of adjacent surfaces strongly indicated that the separation was not due to undercutting of the nickel by corrosion.
3. In areas of normal surface temperature, pores in the nickel plate generally resulted in negligible attack of the aluminum. The pits were spherical in shape with no gross undercutting of the nickel.

Al-Ni Bonding

The initial step in studying methods to improve the aluminum-nickel bond was to devise an adherence test that would be more discriminatory than conventional "bend-testing", be amenable to testing plated fuel element (rather than samples of a specific geometry) and simulate the mechanism(s) causing the in-reactor sloughing. Initial attempts included rapid thermal cycling from >100° C to 15° C by induction heating, and thermal cycling from 500° C and 200° C salt baths to a cold water quench. The chemical-nickel plate developed visible cracks under the most

6. HW-62441, op. cit., pag 2.

severe thermal cycling conditions, but no sloughing of any consequence. Likewise corrosion testing in reactor coolant failed to produce plate sloughing after 22 weeks at 120° C and 18 weeks at 160° C. W. E. Tragert (GERL) suggested that radiolytically generated atomic hydrogen might be a contributing factor, since it diffuses readily through nickel and notably, the rate of diffusion into a void is virtually independent on the pressure of molecular hydrogen therein.⁷ Thus, over a period of time, sufficient pressure might build up and cause blistering and detachment of the plate. The validity of this mechanism was checked by generating atomic hydrogen electrolytically on the surface of nickel plated aluminum, and it was found that the nickel plate could be blistered to the extent that gross sloughing occurred in a few hours. Further examination of irradiated fuel elements revealed blisters which resembled those produced electrolytically. Further evidence was sought by analyzing the gas pumped from the surfaces of nickel-plated irradiated fuel elements. Results from four plated and four unplated elements were inconclusive, with <.01 to 9.5 per cent hydrogen in gas from the nickel plated elements and <.01 to 2 per cent hydrogen in gas from unplated elements.

Considering the apparent dependence of in-reactor sloughing on neutron flux per se, the resemblance between blisters on irradiated fuel elements and those produced by electrolytically generated hydrogen, and the negative results of ex-reactor thermal cycling and corrosion tests, hydrogen absorption appeared to be the most plausible mechanism for the observed in-reactor behavior. One would expect, however, that sloughing would be severest in the maximum flux region, (where radiolysis of the water is maximum) rather than upstream of the flux peak, as illustrated in Figure 1. This apparent anomaly may be due to several factors including: (1) insufficient data (the degree of sloughing as determined at present is highly qualitative, due to the inherent difficulty in examining irradiated fuel elements) and (2) effect of impurities in the water and on the fuel element surface. (Glasstone⁸ states that both impurities and temperature may markedly influence the chemical equilibrium conditions in the radiolytic decomposition of water; these effects would be more pronounced going downstream in a reactor tube.) Thus, blistering of the plate with electrolytically generated hydrogen was the most acceptable adherence test method, since it appeared to reasonably simulate the in-reactor sloughing mechanism, produced a measurable effect in a few hours' time, and could be readily employed for a variety of plated geometries. A testing system was developed by HLO Coatings and Corrosion Operation⁹ and further refined and employed by the writer.

7. "Gases and Metals", C. J. Smithells, (Chapman and Hall Ltd., London, 1937).

8. "Principles of Nuclear Reactor Engineering", S. Glasstone, pages 491-493 (D. Van Nostrand Co., - 1955).

9. HW-61634, "An Investigation of Cathodic Hydrogen Evaluation on Nickel-Plated Aluminum", R. Sorenson, September 3, 1959.

It appeared to be a reasonable assumption that the Al-Ni bond could be improved by baking the plated fuel elements in the 300-500° C temperature range, thus stress-relieving the plate and forming an aluminum-nickel diffusion layer. Based on experience with hot-pressed fuel elements, the Al-Ni diffusion bond has adequate mechanical strength and stability in an irradiation field. While the times and temperatures normally employed in hot-press bonding may be deleterious to the existing Al-Si uranium bond, it also appeared reasonable that a plating could be diffusion bonded to its substrate at shorter times and lower temperatures, due to the comparatively closer proximity of the surfaces, and the fact that a fairly strong bond already exists. Tests covered a temperature range of 300-400° C and times up to 25 hours. Samples were 0.5-mils thick chemical-plate from two vendors, including five separate plating batches. Both "good" and "poor" plate from each vendor is included, the "good" plate having satisfactory as-plated adherence, and the "poor" plate having adherence such that the plate readily detached on bending the base metal. The "good" plate is characteristic of that irradiation tested in PT-IP-207-A. Autoclaved plating was used as a standard, since the fuel elements irradiated in IP-207-A had been autoclaved after plating. Data are represented in Table I, and typical results are illustrated in Figure 2.

TABLE I

BEHAVIOR OF CHEMICAL-NICKEL PLATE IN THE HYDROGEN BLISTERING TESTAutoclaved Only

Poor Plate	60% of surface area blistered (total of 535 in ² incl. 9 fuel elements, 2 cans and 19-1" long can samples)
Good Plate	40% of surface area blistered (total of 660 in ² incl. 9 fuel elements, 4 cans and 31-1" long can samples)

Autoclaved & Baked

Bake Temp., °C	Per Cent of Surface Area Blistered					
	Poor Plate			Good Plate		
Bake Time-Hr.	300	350	400	300	350	400
1/6	---	---	---	78	---	---
1/2	50	50	100	75(2)	8	1
1	2	1	5	5(3)	0	---
2	0	2	---	0(4)	0	0
3	22(1)	---	11	0(5)	0	0
6	29	38	27	0	0	---
12	2	---	0	0	---	---
25	27	---	---	---	---	---

1. 260 in² including 5 fuel elements and 13 can samples 1" long
2. 370 in² including 8 fuel elements and 11 can samples 1" long
3. 50 in² including 11 can samples 1" long
4. 50 in² including 11 can samples 1" long
5. 350 in² including 4 fuel elements, 4 full cans, 7 samples 1" long

Other data from 14-32 sq. in. including samples from 3-7 cans.

The following are evident:

1. The adherence of "poor" plate cannot be consistently improved by baking.
2. The adherence of "good" plate is vastly improved by baking for two hours and longer at 300° C or for one hour and longer at 350-400° C.

Other data indicated that deep alkaline etching of the aluminum prior to plating has no beneficial effect.

The behavior of the "poor" plate is significant in that it supports the erstwhile assumption that the test is indeed a measure of adherence. It is recognized that the tendency to blister may be markedly influenced by surface and sub-surface conditions which may control the absorption and diffusion of atomic hydrogen to a void. If these conditions are controlling, the "poor" plate should behave the same as the "good" plate. Since this is not the case, one must conclude the difference is improved bonding at the nickel-aluminum interface.

Metallographic examination of the interface between aluminum and chemically deposited nickel did not reveal any significant changes resulting from autoclaving and baking for 1-6 hours in the 300-400° C range. However, since visible diffusion layers are not a necessary condition for sound aluminum-nickel bonding,¹⁰ their absence is probably of little consequence.

Effect of Baking on the Al-Si - U Bond

The effect of a post-plating bake treatment on the integrity of the Al-Si - U bond was assessed by measuring loss of tensile strength, ultrasonic testing, and metallographic study. Baking at times and temperatures which produce a significant improvement in Al-Ni adhesion caused no appreciable deterioration in Al-Si - U bond quality.

Statistical analysis of tensile strength data (See Table II) established the following limits of time and temperature for no-loss of tensile strength:¹¹

300° C	350° C	400° C
3.2 hrs.	1.4 hrs.	0.6 hrs.

The average stud pulling force is reduced by about twenty per cent after the following times:

300° C	350° C	400° C
6 hrs.	2.8 hrs.	1.5 hrs.

10. CRMET-703 (AECL), "The Development of Bonded Fuel Elements by the Process of Extrusion Cladding", G. V. Kidson, May, 1957, and personal communication with Dr. A. J. Mooradian (AECL).
11. "Effects of Bond Strength of Heat Treating After Nickel Plating", J. L. Jaech to G. F. Jacky, November 27, 1959.

TABLE II

POUNDS FORCE REQUIRED TO PULL 1/4" DIAMETER STUD

<u>Bake Temp., °C</u>	<u>Time Hr.</u>	<u>Max.</u>	<u>Min.</u>	<u>Median</u>	<u>Average</u>	<u>No. Fuel Elements</u>	<u>Total No. Studs</u>	
No Bake	--	1150	300(2)	1000	860	10	30	Group I (1)
300° C	3	1150	400	1000	910	4	12	
	6	1150	250(3)	875	820	4	24	
	12	950	200	500	530	4	24	
	25	800	200	300	350	4	24	
	50	700	200	300	330	4	12	
350° C	3	1050	450	630	670	4	12	
	6	1100	300	550	580	4	12	
	12	750	150	430	450	4	12	
400° C	1/2	1300	350	950	860	4	24	
	1	1200	350	800	790	4	36	
	3	850	400	600	630	4	12	
	5	975	90	450	500	4	24	
	22	500	200	350	340	4	12	
No Bake	--	1050	300(4)	700	680	10	30	Group II (1)
300° C	3	1050	200(5)	730	680	10	30	

1. Elements in a single group canned on same day
2. Three studs at < 400 lbs.
3. Two studs at < 400 lbs.
4. Six studs at < 400 lbs.
5. One stud at < 400 lbs.

Metallographic examination¹² generally confirmed results of tensile testing. Additional UAl_3 was formed during baking at 300° C and 350° C for three hours, 400° C - 1/2 hr., and increased with time at these temperatures. Since UAl_3 is one of the more ductile compounds formed during canning, increased quantities may toughen the bond with some loss in tensile strength. No additional diffusion of uranium into the Al-Si braze layer was observed.

Ultrasonic testing indicated no adverse effects of baking at 300° C up to six hours, providing good bonding was achieved in canning. Twenty-two elements from two canning periods showed no increase in total count after baking for three hours

¹². Personal Communication - R. H. Todd (HLO).

at 300° C, and twelve baked for an additional three hours yielded the same result. The standard bond tester yielded less encouraging results; one element out of the twenty-two was rejected after three hours at 300° C, and two more were rejected after a total six hours at 300° C. However, all three were marginal as indicated by initial total counts 1-1/2 to 2 fold higher than the highest total count in the non-reject population. Stripping revealed non-wet can areas 1/4 in. to 3/8 in. diameter, which probably showed up as a void after mild thermal cycling.

Coolant Flow Restriction

Flow tests indicated that it is highly unlikely that sloughed chemical-nickel plate would significantly reduce coolant flow in a reactor-process tube. Tests were carried out in a BDF tube mock-up at a flow rate of ~31 gpm. The charge consisted of the normal downstream dummy pattern, twenty-four I & E fuel elements and, upstream, eight I & E fuel elements, each having approximately 2 sq. inches of 2-3 mils thick chemical nickel plate attached to the surface with water-soluble glue. Individual pieces of nickel ranged from approximately 1/4 in. x 1/4 in. to 1 in. x 1 in. in size, with at least one large piece on the external surface in each fuel element, and an approximate 1/4 in. wide x 1 in. long piece in the bore. Pressure measurements included front header-to-venturi ΔP , venturi pressure (by Pannellit gage) and rear pigtail ΔP . The initial test was run for 45 minutes and no significant ΔP variations or Pannellit surges were observed or recorded. Examination of the fuel elements revealed that 1/2 to 2/3 of the nickel had been sloughed. Pieces as large as 5/8 in. long x 1/4 in. wide were found still clinging to some of the downstream fuel elements, indicating sloughing was a gradual process--and not an initial burst which could conceivably occur before steady state conditions were established. A second test of two hours duration with the same fuel elements removed the remainder of the nickel and again no indication of a flow restriction was observed. About one sq. in. of plate had accumulated annular flow groove of the outlet nozzle. Since the pigtail connection was topside, conditions were optimum for accumulation; the fact that over ninety per cent of the sloughed plate was washed out indicates little or no chance of an accumulation and resultant flow restriction in this region. These results are particularly significant, considering that the plate used was 2-3 mils thick compared to the 0.5 mils or less considered for in-reactor service.

G. F. Jacky
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FIGURE 1

WEIGHT-LOSS AND NICKEL-SLOUGHING DATA

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(a) "Poor" Plate - Autoclaved

(b) "Good" Plate - Autoclaved

(c) "Good" Plate - Autoclaved and Baked
3 hrs. at 300°C.

FIGURE 2

Typical Behavior in the Electrolytic Hydrogen
Test

Note: Samples (b) and (c) are from same can. Plate is nominal
0.5 mils thick, chemically-deposited.

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