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TITLE

THE PREPARATION AND IRRADIATION BEHAVIOR OF CHEMICALLY-NICKEL PLATED ALUMINUM-JACKETED FUEL ELEMENTS

(A Paper for Presentation at the 14th Annual Joint U.S.-Canadian Sheath Conference)

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THE PREPARATION AND IRRADIATION BEHAVIOR OF CHEMICALLY-

NICKEL PLATED ALUMINUM-JACKETED FUEL ELEMENTS

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INTRODUCTION

Nickel plated aluminum was considered as a jacketing material for nuclear fuel elements as early as 1954, and both static and dynamic corrosion tests were carried out by Argonne National Laboratories⁽¹⁾ and by Atomic Energy of Canada, Ltd.,⁽²⁾ employing demineralized water at temperatures of from 260 to 316° C. Results generally indicated that the nickel had excellent corrosion resistance; however, difficulties were experienced in achieving satisfactory continuity and adhesion of the plate; subsequent work emphasized Ni-Aluminum alloy development. At Hanford, our earliest experience (1957) employed Ni plate on aluminum-jacketed fuel elements primarily to minimize mechanical damage to the jacket surface during an irradiation test. The appearance of these fuel elements after discharge suggested that the nickel plate might also be a highly satisfactory coating for corrosion and abrasion resistance. Incentives were manifold, including reducing the incidence of in-reactor fuel element failures and permitting reduction of the aluminum jacket thickness with a concomitant increase in space available for uranium or for cooling water passage. A program has been carried out for the past three years aimed at determining various methods of employing nickel plated aluminum jacket material and testing the capabilities of high quality commercial-grade plate. Almost exclusively chemically deposited ("electroless")⁽³⁾ plate has been tested, primarily because of the excellent plate-thickness control inherent in the process compared to electrodeposition. This feature was particularly attractive since the fuel

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elements are 9-inch long hollow cylinders, internally and externally cooled,⁽⁴⁾ and a coating of uniform thickness on all coolant-contacting surfaces is desirable. To minimize neutron capture by nickel, the plate was applied to fuel elements jacketed in C-64 alloy aluminum,⁽⁵⁾ which contains no nickel as an alloying element.

The initial approach to utilizing nickel-plated aluminum was plating of the finished fuel element. The plating has for the most part been done by commercial platers in the Seattle area. Development and testing work included an irradiation test to determine the effect of nickel plate on contamination of fuel element cooling water, development of methods to test and improve adherence of the nickel plate, and corrosion testing plated fuel elements, both in-reactor and ex-reactor. Results to date indicate that chemically deposited nickel plate 0.5 mils (.0005 inch) thick has a high potential for increasing the corrosion resistance of aluminum jacketing material in the environment of the reactors presently operating at Hanford, and testing is being continued and expanded.

A second approach toward employing nickel-plated aluminum jacketing material is plating of the aluminum components prior to assembling the fuel element by the braze-dip canning method. This approach is less straightforward than plating the finished fuel element, involving changes to the normal assembly process cycle. A feasible assembly process cycle has been developed.

The writer wishes to acknowledge the assistance of P. L. Lee in carrying out much of the experimental work described herein.

EXPERIMENTAL WORK AND RESULTS

The initial irradiation test was carried out to determine the effect of nickel plate on the radioactivity and radioactive elements present in the reactor cooling water. This information was essential since the reactor effluent is

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discharged into the Columbia River, and an intolerable increase in the radio-activity level due to the nickel plate would, of course, preclude its use. Possible contaminants included Ni^{63} , Co^{58} , Co^{60} , and P^{32} (radio-phosphorus is produced by transmutation of the 8-10 wt percent natural phosphorus present in the chemically-deposited nickel plate). The test included four reactor tubes each containing 32 Hanford standard fuel elements. Two tubes were plated by electrodeposition, and the remaining by chemical deposition. The four tubes were irradiated to an integrated exposure of approximately 400 MWD/ton. Periodic samples of the effluent stream from each tube did not indicate that effluent contamination would be a problem, although the data were not conclusive. A second encouraging result of the test was excellent corrosion resistance exhibited by 0.5 mil (.0005-inch) thick plate in a severely overheated area on the fuel element surface. On the other hand, approximately a third of the fuel elements showed evidence of plate sloughing, and on some of these up to 1/4 of the aluminum surface was exposed. Thus, the test revealed potentiality for prolonging fuel element life; but a significant improvement in plate adhesion was essential.

Improving the plate adhesion was a twofold problem (1) determining the cause of the plate sloughing and developing remedial measures, (2) developing a plate adhesion test which would adequately discriminate. (The widely-used bend test indicated the plate used in the irradiation test described above had good adhesion.) Initially it was thought that sloughing of the nickel was due to either or both thermal cycling and corrosion along the aluminum-nickel interface. However, this belief was not confirmed by results of fuel element examinations, described below.

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Weight-loss data and visual and metallographic examination of the fuel elements revealed:

1. The severest nickel sloughing generally occurred in regions of high neutron flux, but upstream of the maximum flux region. This indicated a dependence on neutron flux but not on mechanical strain of the fuel element (which would maximize with flux).
2. Nickel sloughing was notably reduced in the sections of the fuel element column having the highest surface temperature, indicating that corrosion of the underlying aluminum was very probably not a factor causing detachment of the nickel plate. Likewise, in a grossly overheated area ("hot-spot") the nickel plate was in good condition with no evidence of cracks, blisters, or sloughing.
3. Some barely-visible blisters in the plate were observed. Sectioning these revealed that the contours of the plate and the basis metal still matched, and no aluminum corrosion product was present.

Attempts to reproduce the observed sloughing in out-of-reactor tests included rapid thermal cycling from $>100^{\circ}\text{C}$ to 15°C by induction heating, and thermal cycling from 500°C and 200°C salt baths to a cold water quench. The plate developed visible cracks under the most severe thermal cycling conditions, but no sloughing of any consequence.

A third possible cause of the nickel sloughing was radiolytically generated atomic hydrogen.⁽⁶⁾ Atomic hydrogen diffuses readily through nickel and notably, the rate of diffusion into a void such as might be present at the aluminum-nickel interface, 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 mechan-

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ism was checked by generating atomic hydrogen on the surface of nickel plated aluminum by electrolysis, 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 by electrolysis. 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 thermal cycling tests, hydrogen absorption appeared to be the most plausible mechanism for the observed in-reactor behavior. An adhesion test was developed, employing the mechanism of blistering the plate by electrolytically generated hydrogen. Briefly, this test is carried out by thoroughly cleaning the surface of the nickel-plated aluminum, then making it cathodic in a slightly corrosive aqueous electrolyte. Poorly adherent nickel will severely blister and spall after as little as 30 minutes at a current density of 6 amp./sq. ft. Comparative evaluation of samples is based on the percentage of the surface area which has blistered after being exposed for a standard time at a standard current density.

Considering methods to improve nickel adhesion, it appeared to be a reasonable assumption that the aluminum-nickel bond could be strengthened 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 pressure-bonded fuel elements, the Al-Ni diffusion bond has adequate mechanical strength and stability in an irradiation field. Baking tests covered a temperature range of 300-400° C and times up to 25 hours. Samples were cylindrical coupons of 0.5-mils (.0005-inch) thick chemically deposited nickel plate, having both "good" and "poor" adhesion in the

as-plated condition. The bond between the "poor" plate and basis metal was such that the plate could be readily detached by bending the coupon. Plate which had been autoclaved 16-20 hours in 90-95 psig steam was used as a standard, since the material which had sloughed badly during irradiation had been given this treatment. Behavior in the hydrogen blistering test is presented in Table I.

TABLE I

BEHAVIOR OF CHEMICAL-NICKEL PLATE IN THE HYDROGEN BLISTERING TEST

Autoclaved Only⁽¹⁾

"Poor" Plate	60% of surface area blistered (total of 535 in ² tested)
"Good" Plate	40% of surface area blistered (total of 660 in ² tested)

Autoclaved⁽¹⁾ and Baked

Percent of Surface Area Blistered

<u>Bake Temp., °C</u> <u>Bake Time-Hr.</u>	<u>"Poor" Plate</u>			<u>"Good" Plate</u>		
	<u>300</u>	<u>350</u>	<u>400</u>	<u>300</u>	<u>350</u>	<u>400</u>
1/6	---	---	---	78	---	---
1/2	50	50	100	75(3)	8	1
1	2	1	5	5(4)	0	---
2	0	2	---	0(5)	0	0
3	22(2)	---	11	0(6)	0	0
6	29	38	27	0	0	---
12	2	---	0	0	---	---
25	27	---	---	---	---	---

- (1) 16-20 hours in 90-95 psig steam
 (2) 260 in² tested
 (3) 370 in² tested
 (4) 50 in² tested
 (5) 50 in² tested
 (6) 350 in² tested
 Other data from 14-32 sq. in.

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The following are evident:

1. The adherence of "poor" plate was not consistently improved by baking.
2. The adherence of "good" plate is vastly improved by baking for one hour and longer at 300-400° C.

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 observed difference is due to improved bonding at the nickel-aluminum interface. Typical results are illustrated in Figure I.

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⁽⁸⁾ their absence may be of little consequence.

It was also essential to consider the effect of any baking treatment on the strength and continuity of the existing bond between the jacket and the uranium core. The bonding material is aluminum-silicon alloy, and the intermediate layers formed with the uranium are affected by prolonged exposure to high temperatures. The effect of bake treatment on the integrity of the jacket-uranium 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 aluminum-nickel adhesion caused no appreciable bond deterioration. Statistical analysis of bond tensile-strength data established that no

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loss in tensile strength occurred after baking for 3.2 hours at 300° C and 0.6 hours at 400° C. The tensile strength was reduced by about twenty percent after six hours at 300° C and 1.5 hours at 400° C. A baking temperature of 300° C was selected for fuel elements to be subsequently irradiation tested, since a wide range in time could be employed, and highly specialized heat treating equipment would not be required.

A second irradiation test was designed to determine if the 300° C post-plating bake treatment indeed produced a significant improvement in nickel adhesion as indicated by the electrolytic-hydrogen blister test. The test included ten reactor tubes, each containing seventeen standard production internally and externally cooled fuel elements clad in X-8001 alloy aluminum,⁽⁹⁾ alternated with fuel elements of the same geometry clad in C-64 alloy aluminum and having a 0.5-0.7 mils (.0005-.0007-inch) thick chemically-deposited nickel plate. The nickel plated fuel elements in six of the tubes were baked for three hours at 300° C following plating, and the remaining four tubes for six hours at 300° C. One tube of each was discharged at approximately 550 MWD/T integrated exposure, and visual examination disclosed no evidence of nickel sloughing. The remaining six charges were irradiated to approximately 800 MWD/T integrated exposure. Visual examination and in-reactor weight loss data disclosed that virtually 100% of the nickel plate adhered. Possible sloughing of areas 1/4 inch or less in diameter on a few fuel elements was reported; however, careful examination of some of these indicated the nickel was probably still present. No difference was observed between plate baked for three hours and six hours. Thus it was successfully demonstrated that 0.5 mil (.0005 inch) thick nickel plate properly selected and processed will adhere to aluminum for attractively long periods of time.

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Additional results of the two irradiation tests described above and other out-of-reactor tests included encouraging information on the degree of corrosion protection afforded by the nickel plate. Weight loss of fuel elements irradiated to 750 to 800 MWD/T integrated exposure, indicated that the corrosion of the nickel plated fuel elements was so low as to be virtually immeasurable. Data comparing nickel plated and X-8001 alloy-jacketed unplated fuel elements are presented in Figure 2. These data were obtained from eight reactor tubes of fuel elements with nickel plated and unplated elements in successive positions in each tube. It is evident that in the highest corrosion zone, the uniform corrosion of the nickel is approximately zero, while the X-8001 weight loss was significantly higher, ranging from 3 to 9 gm. The increased weight loss of nickel plated fuel elements in the upstream portion of the tube cannot be explained at present. The aluminum corrosion rate in this region is very low; visual examination disclosed no sloughing of the plate or other phenomena which might be accountable. It was observed that the "crud" film deposit on nickel plated fuel elements weighted less than that on X-8001 alloy aluminum surface by a factor of approximately 2-1/2. Since the crud film significantly impedes transfer of heat to the coolant, its reduction would very probably reduce the jacket surface temperature.

Visual and metallographic examination of several fuel elements having severely overheated surfaces (probably due to misalignment in the horizontal process tubes) revealed that a 0.5 mil (.005-inch) thick nickel plate was highly effective in preventing corrosion of the underlying aluminum. The most severe "hot spot" was conservatively estimated to be at a temperature of over 300° C for a period exceeding 100 hours, based on the appearance of the underlying aluminum-silicon braze material, and the extent of aluminum nickel diffusion. A heavy frangible boiler scale had been deposited on the fuel element surface; however, the underlying nickel was shiny and adherent. Sectioning revealed no corrosion

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of the aluminum except at the edges of the "hot spot" region, where the nickel appeared to be poorly adherent (this fuel element was from the initial irradiation test, wherein some of the fuel elements sloughed up to 25 percent of the nickel). Photographs of the "hot spot" and metallographic sections are presented in Figures 3 and 4. Other less severe "hot spots" were visually and metallographically examined with no evidence of corrosion of the underlying aluminum.

A possible deficiency in the chemically deposited nickel plate was revealed by the presence of numerous hairline cracks generated in regions of highest neutron flux. These cracks were observed on approximately 15 percent of the 170 nickel plated fuel elements irradiated to 800 MWD/T. They were generally circumferential and thus appeared to be the result of the cyclic straining of the fuel element (Figure 5). They occurred in greatest abundance in the cooler surface areas of a single fuel element (in the vicinity of the supporting process tube ribs) and were notably absent in "hot spot" areas. A single fuel element was visually and metallographically examined and measured. No dimensional distortion was evident that would account for the cracking. Examination of metallographic sections of several cracks revealed that aluminum corrosion was negligible (up to $\sim 1/2$ mil) and that the cracks did not propagate into the aluminum. Typical sections are presented in Figure 6. Thus, it was concluded that while this tendency to crack was undesirable, it should not preclude further testing of the material.

Other corrosion testing included assessment of possible accelerated corrosion of the aluminum at discontinuities in the nickel plate. Fuel elements with the nickel plate deliberately defected were exposed up to 9 months in out-of-reactor flow tubes, using reactor cooling water heated to temperatures of

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120° and 165° C. Visual examination revealed no accelerated aluminum corrosion at discontinuities in the nickel plate. In general, aluminum corrosion appeared to be equivalent or less than for unplated material. Typical sections are presented in Figure 7. It was found, however, that the aluminum will corrode rapidly at discontinuities in the nickel plate in some aqueous environments. Penetration rates on the order of 5-6 mils (.005-.006-inch) per week were experienced on some fuel elements immersed in the water of our storage and examination basins. This water contains, among other ions, small amounts of aluminum deoxidizing compounds.

Concurrent with the testing of fuel elements plated on the coolant contacting surfaces after normal assembly and finishing, a process has been developed using aluminum components which are nickel plated on all surfaces, prior to assembly by the braze-dip method. A singular advantage of this fuel element is that a double layer of nickel (on both the internal and external surface of the jacket) is provided for corrosion protection -- thus a discontinuity in one layer would probably not be reproduced in the second in the same location and the over-all integrity of the jacket would not be seriously impaired. A second advantage is that dissolution of the aluminum jacket by the molten aluminum-silicon braze material can be eliminated by the barrier of nickel plate. A third process advantage is that the braze-dip assembly process provides a heat treatment which appears to be highly effective in assuring adhesion of the nickel plate, since a thin aluminum-nickel diffusion layer is produced. Thus far, the process appears capable of producing fuel elements equal or superior in quality to our normal Hanford production, based on destructive and non-destructive testing of small pilot-scale quantities. The process involves minor modifications to the normal assembly cycle, the equipment, both the uranium core and aluminum

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jacket designs, and the closure welding process. Corrosion tests include a three month exposure to 175° C tap water (HAPO, 300 Area - a fairly corrosive medium) for eleven fuel elements. Visual examination disclosed no corrosion of the weld beads or other exposed aluminum. Irradiation testing has not yet been carried out.

SUMMARY

Feasible processes for testing and improving adhesion of commercial-grade chemically deposited nickel plate on aluminum have been developed. Plate adhesion testing is based largely on resistance to blistering during exposure to electrolytically generated atomic hydrogen. Adhesion of high-quality plate is markedly improved by baking at 300-400° C for periods of one hour and longer. The adhesion of high-quality 0.5 mils (.0005-inch) thick chemically deposited plate baked for 3-6 hours at 300° C appears to be adequate for exposure up to 800 MWD/T as indicated by small scale irradiation tests. These tests also indicate that 1/2 mil (.0005-inch) thick plate is highly effective in protecting the aluminum from both uniform corrosion and accelerated corrosion in overheated areas. Indeed, the behavior of the nickel plate appears to improve as the surface temperature increases. Corrosion of the aluminum due to galvanic action at discontinuities in the nickel plate has not been observed in Hanford reactor cooling water. Contamination of the cooling water with radioactive materials generated in the nickel plate does not appear to be a problem; however, the present data are not conclusive. While the experience cited above has been developed using fuel elements plated on the coolant-contacting surfaces only, a probably-improved model has been developed, employing aluminum components which are nickel plated on all surfaces, prior to the braze-dip assembly process. Advantages include a double layer of nickel which should provide improved resis-

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tance to water penetration, compared to a single layer, and a heat treatment provided in the braze-dip assembly process which appears to be highly effective in improving adhesion of the nickel plate.

FUTURE WORK

In view of the excellent corrosion resistance exhibited by 0.5 mil (.0005-inch) thick chemically deposited nickel plate on C-64 alloy aluminum, irradiation testing of fuel elements plated on the coolant contacting surfaces will be expanded in scope and severity. At the time of the writing, a test including 1800 fuel elements having a 0.5-mil (.0005-inch) thick plate and 320 fuel elements having a 0.2-mil thick plate has been prepared and awaits a scheduled reactor shutdown for charging. Irradiation to ~1400 MWD/T is planned. The purpose of this test is to obtain more reliable information on the adherence of the nickel plate, indicate the failure resistant capabilities of the fuel element, and provide additional data on effluent contamination. A second fairly large scale irradiation test is being planned, which will include approximately 5000 fuel elements with uranium cores enriched to 0.94 percent, jacketed in C-64 alloy aluminum and plated with 0.1-0.2 mils (.0001-.0002 inch) thick chemically deposited plate. An irradiation test of approximately 200 fuel elements jacketed in aluminum components which have been nickel plated prior to braze-dip assembly is planned for charging within the next few months pending final results on fuel element quality testing.

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1. ANL 5461 - "Reactor Engineering Division Quarterly Report on Power Reactor Programs", January 1, 1955 through March 31, 1955.
2. CR-MET-756 (AECL) - The Corrosion Resistance of Nickel-Plated Aluminum Nickel Alloys in High Temperature Water, G. J. Bieffer, 5-8-58.
3. Properties of chemically-deposited ("electroless") nickel are presented in "Symposium on Electroless Nickel Plating", ASTM Special Technical Publication No. 265.
4. This fuel element is a thick-walled hollow cylinder, 9 inches long x 1.44 inches OD with an $\frac{3}{8}$ inch I.D. axial cooling channel.
5. Nominal composition of C-64 alloy aluminum is:

Aluminum	99.3 wt % (min.)
Iron	0.3-0.5 wt %
Silicon	0.17 wt % (max.)
6. Suggested by W. E. Tragert (General Electric Research Laboratory).
7. "Gases and Metals", C. J. Smithells (Chapman and Hall, Ltd., London, 1937).
8. 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).
9. Nominal composition of X-8001 alloy aluminum is:

Aluminum	97.5 wt % (min.)
Nickel	0.9-1.30 wt %
Iron	0.45-0.70 wt %
Silicon	0.17 wt % (max.)
Copper	0.15 wt % (max.)

(a) "Poor" Plate - Autoclaved (20 hrs in 95 psig Steam)

(b) "Good" Plate - Autoclaved

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

FIGURE 1

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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FIGURE 3

Hot Spot-1/2 mil Thick Chemical-Nickel Plate on C-64 Alloy Aluminum.
Surface Temperature Estimated at 300-325 C >100 hrs.

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a. Maximum Temperature Region

b. Nickel
 Ni-Al Diffusion Layer
 Aluminum

c.

d. Fringe Region

FIGURE 4
Effect of High Surface Temperature on Ni-Al Interface
(Sections of hot-spot shown in Fig. 3)

FIGURE 5

Typical Circumferential Cracks in Nickel Plate of an Irradiated Fuel Element (White deposits are "Barnacles" from corrosion in storage basin water)

A.

500 X

B.

Nickel

Aluminum

C.
2000 X

Note excellent conformation of plate to substrate

FIGURE 6

Typical Sections of Cracks Shown in Fig. 5

Note negligible corrosion of aluminum and absence of crack propagation into the aluminum

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- a. Can Wall Section at 250X. Nickel was deliberately defected with a punch, then exposed to a 165 C process water tube for 5 1/2 months.
Note: Presence of Nickel plate on side and bottom of hole.
Maximum depth of corrosion is approximately 2 mils.

- b. Nickel was defected by filing off an approximate 1/8 inch wide strip, then exposed in 120 C process water flow tube for 5 1/2 months.
Maximum corrosion depth approximately 7 mils.

FIGURE 7

Corrosion of Aluminum at Discontinuities in Nickel
(Flow Tube Tests)

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