ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

COMPATIBILITY OF CHROMIUM PLATING
WITH THE EBR-II COOLANT

by

W. E. Ruther and H. A. Taylor*

EBR-II Project

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

April 1973

This document is
PUBLICLY RELEASABLE

Mary R. Hale, ANL
Authorizing Official
Date 8-17-09

MASTER

*Hot Fuel Examination Facility, Argonne-West

Reproduction for Announcement in Nuclear
Science Abstracts Distribution Limited
to Participants in the EBR Program.
Others request from TIC.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

.

.

.

.

.

.

TABLE OF CONTENTS

ABSTRACT	1
I. INTRODUCTION	1
II. PROCEDURE AND EVALUATION	2
A. Examination of Hardware from Reactor	2
B. Experimental Irradiation	3
III. DISCUSSION AND CONCLUSIONS	8
REFERENCES	8

LIST OF FIGURES

1. Reactor Grid Plates and Lower Adapters of Subassemblies	1
2. Solubility of Chromium in Sodium	2
3. Mark-P1 Irradiation Subassembly	3
4. Top Portion of Stack of Chromium-plated Rings from Subassembly X102	6
5. Bottom Portion of Stack of Chromium-plated Rings from Subassembly X102	6
6. Comparison of As-plated and Irradiated Chromium Plating Applied by "Electrolizing" Process and Standard Electroplating Process	7
7. As-plated and Irradiated 450–550 μ in. Chromium Plating Applied by Standard Electroplating Process.	8

LIST OF TABLES

I. Chromium Thickness on Exposed Outside Surfaces of Lower Adapters	3
II. Order of Stacking Specimens, and Weight Change after Exposure	4
III. Irradiation History of Experimental Subassembly X102	5
IV. Weight Change for Plating Type and Thickness	5

COMPATIBILITY OF CHROMIUM PLATING WITH THE EBR-II COOLANT

by

W. E. RUTHER AND H. A. TAYLOR

ABSTRACT

The compatibility of chromium plating with the EBR-II coolant was determined by two techniques. First, stainless steel components that had been chromium-plated were examined after years of exposure to primary sodium in the reactor. Second, a special subassembly was exposed in which a loss of chromium plating of as little as 10^{-6} in. could be detected by gravimetric means. In this subassembly, specimens plated to different thicknesses by a standard commercial electroplating process and by a proprietary "Electrolyzing" process were exposed to EBR-II primary sodium for 13 months. The results obtained by both techniques indicate that chromium plating is completely compatible with the flowing EBR-II coolant at temperatures at least as high as 845°F.

I. INTRODUCTION

The compatibility of chromium plating with the EBR-II coolant is of great interest, because several parts of the reactor were designed to utilize the properties of such plating. For example (see Fig. 1), the lower adapters of all subassemblies were constructed to be inserted into close-fitting holes in the Type 304 stainless steel reactor grid plates. Insertion and withdrawal of these subassemblies, which may take place at temperatures up to 700°F in the sodium pool, created a potential galling and self-welding problem. The possibility of galling was increased also in the sodium by the absence of the usual oxide (air) temper film on the contacting stainless steel members. The designer's solution, which has worked out well in practice, was to chromium-plate the lower adapter, which is replaceable. The plating did not have to undergo heavy wear, such as that imposed by several hundreds of sliding operations. However, enough of it had to remain to ensure that a surface film of chromium would separate the stainless steel parts for the life of the subassembly.

Fig. 1. Reactor Grid Plates and Lower Adapters of Subassemblies (all dimensions in inches)

At the time of the original design, over 15 years ago, no quantitative data were available for estimating a dissolution allowance for the chromium plating, so an arbitrary plating-thickness specification of 0.5–3.5 mils was set initially. This is a relatively thick chromium plating, as compared with those used in other nonwear applications. Unfortunately, the close dimensional tolerances specified for the lower adapter made it necessary to finish-grind after plating, a procedure that added significantly to the cost of the adapter.

Recent measurements of the solubility of chromium in sodium (see Fig. 2) have indicated relatively low values and a strong dependence of the solubility on sodium temperature. A crude extrapolation of these high-temperature data to 371°C (700°F) suggests that the chromium solubility would be of the order of 0.1 ppb at the temperatures of the lower adapter in EBR-II. Since the plating is at approximately the lowest temperature of the primary circuit (except for the cold trap), the potential for temperature-driven mass transport is very low. Therefore, metal-loss rates for the plating should be extremely low, permitting the use of much thinner plating. The thin plating, in turn, would not require the extra finish-grinding operation to meet dimensional tolerances.

Fig. 2. Solubility of Chromium in Sodium¹

Experimental verification of the dissolution rate of the plating by examining chromium-plated hardware with years of exposure in the primary sodium was of limited value, because no preirradiation thickness measurements were available. Therefore, an experimental irradiation in the reactor was conducted to accurately measure the loss of chromium plating and to determine the adherence of very thin platings under reactor conditions.

II. PROCEDURE AND EVALUATION

A. Examination of Hardware from Reactor

The first data available on the performance of chromium plating on stainless steels for EBR-II were obtained from postirradiation sectioning of hardware from the reactor.

The lower adapters from safety-rod thimble 3D1 (2110 days in reactor grid) and from core subassembly C-2027 (366 days in reactor grid) were examined by R. V. Strain; the results are shown in Table I. Note that at one spot, on C-2027, the plating was slightly thinner than the minimum of the preirradiation specification of 0.0005–0.0035 in.

TABLE I. Chromium Thickness on Exposed Outside Surfaces of Lower Adapters

	Thickness, in.
Safety-rod thimble 3D1	
Near flow-orifice holes	0.00045
1 in. above "J" slot	0.00055
Core subassembly C-2027	
Top	0.00050
Near flow-orifice holes	0.00037
SURV-I subassembly	
Near flow-orifice holes	0.0026

The lower adapter from the SURV-I subassembly⁴ was also sectioned after 671 days in the reactor. The plating thickness of 0.0026 in. was within manufacturing specifications.

Finally, the jaw gripper from control-rod driver No. 6 was examined after about 1800 days in the reactor.⁵ This part, manufactured from Type 420 stainless steel, saw sodium temperatures of 830-840°F during reactor operation. A plating thickness of ~ 0.003 in. remained after this exposure. This was within manufacturing specifications.

In each case described, postirradiation metallography revealed a uniform plating thickness within the microscopic field, with imperfections such as microcracks and microfissures and porosity. Since the same sort of imperfections are typical problems in chromium plating, it was not possible to decide whether they were due to the reactor exposure.

B. Experimental Irradiation

The vehicle chosen for the experimental irradiation of chromium plating was a Mark-PI subassembly (shown in Fig. 3). The test specimens were plated Type 304 stainless steel rings approximately 2.2 in. in diameter $\times$ 0.06 in. in wall thickness $\times$ 2.0 in. long. Sodium at a flow rate of 4.6 gpm flowed over the outer surfaces of the rings while stagnant sodium contacted the inner faces. Only the area of the outer face was considered in calculating penetration equivalents of weight loss. The sample size was chosen to permit sensitive and reliable gravimetric measurements on a standard analytical balance.

Fig. 3. Mark-PI Irradiation Subassembly (all dimensions in inches)

Several thicknesses of plating were obtained under Federal Specification QQ-C-320 Type 2, Class 2, from two established commercial vendors Economy Plating, Incorporated, Chicago, Ill., and the Electrolyzing Company, a division of Advance-Wilson Industries, Chicago Heights, Ill. The first vendor applied the plating by a standard electroplating process, the second applied it by his proprietary "Electrolyzing" process. The presence and the approximate thickness of plating were determined by sectioning and polishing a random sample of each batch before the experiment. Similar microscopic examinations were completed on selected samples after irradiation.

The specimens were stacked on the Mark-PI support column in five repeating groups, each of six different ring surfaces. In each group, there was a base Type 304 stainless steel control, three rings plated by one vendor, and two rings plated by the second vendor. The loading sequence is indicated as a part of Table II.

TABLE II Order of Stacking Specimens, and Weight Change after Exposure

Ring Position	Plating Type ^a	Weight Change, mg/specimen
1 (Sodium exit, top)	None	-2.1
2	E-100	+2.5
3	E-200	+3.1
4	S-50	+1.6
5	S-100	+3.2
6	S-500	+2.7
7	None	-2.8
8	E-100	+2.2
9	E-200	+1.3
10	S-50	-0.1
11	S-100	+0.1
12	S-500	+1.3
13	None	-3.0
14	E-100	+0.5
15	E-200	+0.2
16	S-50	-0.9
17	S-100	+2.0
18	S-500	+1.4
19	None	-1.4
20	E-100	+2.3
21	E-200	+0.7
22	S-50	-0.6
23	S-100	-0.2
24	S-500	+1.6
25	None	-0.7
26	E-100	+1.4
27	E-200	+0.7
28	S-50	+0.5
29	S-100	0
30 (Sodium entrance, bottom)	S-500	+0.3

^aE-100 80-150- μ m Chromium plating by "Electrolyzing"
E-200 150-250- μ m Chromium plating by "Electrolyzing"
S-50 20-80- μ m Chromium plating by standard process
S-100 80-150- μ m Chromium plating by standard process
S-500 450-550- μ m Chromium plating by standard process

The maximum temperature reached at the upper adapter of the Mark-PI subassembly was determined by a group of melt-wire indicators. The melt capsule contained duplicate wires of the following types and melting points:

Alloy	Melting Point, °F
Zn-Sb	941
Ag-Sb	900
Au-In	845

Postirradiation examination established that the maximum temperature was between 845 and 900°F.

Sodium was removed from the irradiated subassembly by the standard washing procedure using moist gas and deionized water. Five rinses were used to ensure complete sodium removal under the rings.

The subassembly was dismantled by carefully removing the outer hexagonal can, thus exposing the stacked rings. The rings were then removed easily by sliding them off the support column with clean manipulators. They were then individually packaged to avoid damage in transfer to another hot cell for examination and weighing.

The specimens were weighed on the Christian-Becker Torbal balance that was used to obtain their initial weights. The balance is accurate to ± 0.3 mg, so the uncertainty in the weight changes is $< \pm 0.6$ mg. For the specimens used, a loss of 1.6 mg of chromium corresponds to a penetration of only 1 μm . (0.025 μm), so the slight uncertainty in weighing is inconsequential.

Table III gives the irradiation history of the subassembly, designated X102. No difficulty in handling or abnormal reactor conditions occurred during its irradiation.

TABLE III Irradiation History of Experimental Subassembly X102^a

Date experiment was received at EBR-II	5/10/70
Date installed in reactor grid	2/22/71
Grid position No	7F1
Date removed from reactor grid	3/1/72
Total MWd accumulated	11,224
Calculated fluence at reactor midplane, nvt	1.7×10^{22}
Total days in sodium	394
Reactor power, MWt	62.5
Inlet sodium temperature, °F	700

^a Compiled by W. J. Larson

Visual examination of the exposed specimen rings showed them to be in excellent condition, although some were slightly tarnished as compared with their luster as received. This staining was more pronounced near the top of the specimen stack, as shown in Figs. 4 and 5. The brighter rings are the unplated stainless steel control specimens.

Under a low-power microscope, the stained appearance was revealed as a composite of much smaller individual stain areas. No visible coating was detected by probing. Ultrasonic treatment in distilled water did not remove the stains.

Since the rings were machined to the same dimensions, it is convenient to compare the weight changes in milligrams per specimen. Table II lists the weight changes as a function of position; Table IV rearranges the data in terms of the plating varieties. Only the bare stainless steel specimens consistently lost weight. The maximum loss occurred in the core region and corresponded to a uniform penetration of only 4.3 μm . Most of the plated specimens gained a small amount of weight. However, three of the five specimens plated to a thickness of 20–80 μm lost small amounts of weight. Nevertheless, the plating appeared to be intact when a section of one ring was polished and examined at 650X magnification.

TABLE IV Weight Change for Plating Type and Thickness

Type	Weight Change, mg per ring ^a	Type	Weight Change, mg per ring ^a
No plating	-2.1	S-50	+1.6
Bare Type 304	-2.8	20–80- μm chromium plating	-0.1
stainless steel	-3.0	by standard process	-0.9
	-1.4		-0.6
	-0.7		+0.5
E-100	+2.5	S-100	+3.2
80–150- μm chromium plating	+2.2	80–150- μm chromium plating	+0.1
by "Electroplating"	+0.5	by standard process	+2.0
	+2.3		-0.2
	+1.4		0
E-200	+3.1	S-500	+2.7
150–250- μm chromium plating	+1.3	450–550- μm chromium plating	+1.3
by "Electroplating"	+0.2	by standard process	+1.4
	+0.7		+1.6
	+0.7		+0.3

^a In each grouping of five rings shown, the rings are listed in order from top to bottom of stack.

It was assumed that the very small weight gains of most specimens were due to metallic deposits in some of the surface irregularities of the rings. Such deposits would not be expected to wash off in the normal treatment of the specimen. One ring, the uppermost plated specimen, was ultrasonically cleaned briefly in

Fig. 4. Top Portion of Stack of Chromium-plated Rings from Subassembly X102.

Fig. 5. Bottom Portion of Stack of Chromium-plated Rings from Subassembly X102.

distilled water. Its weight gain was reduced from 2.5 mg (as irradiated) to 1.1 mg by this treatment, tending to substantiate this hypothesis.

Metallography of the irradiated specimens was used only to determine the condition of the plating. The thicknesses of the plating before and after irradiation should not be compared directly, because plating tolerances are large enough to permit significant differences among different specimens. In general, on the basis of the limited metallography, the specimens plated by the standard electroplating process had fewer breaks and cracks in the plating than those plated by the Electrolyzing process. (See Fig. 6) Even the thicker (450–550- μ m) plate put down by the standard process had few defects, as shown in Fig. 7.

Fig. 6 Comparison of As plated and Irradiated Chromium Plating Applied by "Electrolyzing" Process and Standard Electroplating Process (Mag ~700X)

As Plated

Irradiated

Fig. 7. As-plated and Irradiated 450–550- μ in. Chromium Plating Applied by Standard Electroplating Process. (Mag $\sim 700\times$)

III. DISCUSSION AND CONCLUSIONS

The chromium platings tested in subassembly XI02 were completely compatible with EBR-II primary coolant for the 13-month exposure at temperatures up to at least 845°F. Examination of reactor hardware having much longer exposures confirmed this experimental result.

Coatings applied by the standard electroplating process were slightly superior in the appearance of the microscopic cross section to those applied by the propriety "Electrolizing" process, but both platings were excellent in their resistance to sodium.

The very thin (20–80- μ in.) plating was difficult to inspect in cross section. Therefore, securing adequate and consistent assurance of its quality might be difficult. The weight changes for this thickness also showed slight abnormalities, compared with those for heavier platings.

It is feasible and desirable to consider reactor use of 100- μ in. (0.1-mil) plating for those parts that do not fret in services or require a capacity for repetitive wear.

REFERENCES

1. T. D. Claar, *Solubilities of Metallic Elements in Liquid Sodium*, Reactor Technol. 13(2), 127 (1970).
2. R. M. Singer, A. H. Fleitman, J. R. Weeks, and H. S. Isaacs, "Measurements of the Solubility of Iron and Chromium in Sodium," in *Corrosion by Liquid Metals*, J. E. Draley and J. R. Weeks (Eds.), Plenum Press, NYC (1970).
3. R. L. Eichelberger and R. L. McKisson, "The Solubility of Cu, Mo, Nb, Fe, Ni, and Cr in High-purity Sodium," *Proceedings of the International Conference on Large Fast Reactor Design*, November 7-9, 1968, ANL-7520, Part I, pp. 319-324.
4. S. Greenberg, *The EBR-II Materials-surveillance Program: I. Program and Results of SURV-1*, ANL-7624 (Sept 1969).
5. W. E. Ruther, T. D. Claar, S. Greenberg, and R. V. Strain, *Materials-Coolant Interactions in EBR-II*, ANL-7670 (Mar 1970).