Contract No. W-7405-eng-26

METALS AND CERAMICS DIVISION

COMPATIBILITY STUDIES OF POTENTIAL MOLTEN-SALT BREEDER REACTOR MATERIALS IN MOLTEN FLUORIDE SALTS

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Date Published: May 1977

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
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UNION CARBIDE CORPORATION
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ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

MASTER

CONTENTS

COMPATIBILITY STUDIES OF POTENTIAL MOLTEN-SALT BREEDER
REACTOR MATERIALS IN MOLTEN FLUORIDE SALTS

J. R. Keiser

ABSTRACT

    This report summarizes the molten fluoride salt compatibility
studies carried out during the period 1974—76 in support of
the Molten-Salt Reactor Program. Thermal-convection and forced-
circulation loops were used to measure the corrosion rate of
selected alloys. Results confirmed the relationship of time,
initial chromium concentration, and mass loss developed by
previous workers. The corrosion rates of Hastelloy N and
Hastelloy N modified by the addition of 1—3 wt % Nb were well
within the acceptable range for use in an MSBR.

INTRODUCTION

The purpose of this report is to summarize the corrosion studies
carried out for the Molten-Salt Reactor Program during the period
September 1974 through May 1976. These studies were intended to determine
the corrosion resistance of potential Molten-Salt Breeder Reactor contain-
ment vessel materials in molten fluoride salt. The nickel-base alloy
Hastelloy N was used successfully for the containment vessel of an
experimental molten-salt reactor, the Molten-Salt Reactor Experiment.
However, the discovery of irradiation embrittlement and grain boundary
embrittlement by the fission product tellurium led to a program to
develop an alloy that would be sufficiently resistant to the conditions.
To ensure that any new or modified alloy would have a high resistance
to corrosion by the fluoride salt, salt-metal corrosion studies were
made. Materials investigated include Hastelloy N, chromium and niobium
modifications of Hastelloy N, Inconel 601, and type 316 stainless steel.
The stainless steel was tested in $LiF-BeF_2$ (65-35 mole %), and the other
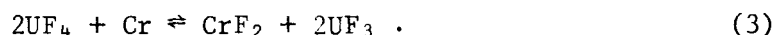alloys were tested in MSBR fuel salt, $LiF-BeF_2-ThF_4-UF_4$
(72-16-11.7-0.3 mole %).

Previous reserachers[1,2] have measured the corrosion resistance of
Hastelloy N and two types of stainless steel in various fluoride salt
mixtures. Their work has shown a multicomponent alloy is corroded by
the oxidation and removal of the least noble component. For Hastelloy N,
the least noble component is chromium. Fluoride salts can oxidize
chromium by reaction with impurities in the salt such as HF, NiF_2, and
FeF_2 and by reaction with constituents of the salt. Impurity reactions
expected are

$$2HF + Cr \rightleftharpoons CrF_2 + H_2 \qquad (1)$$

and

$$FeF_2 + Cr \rightleftharpoons CrF_2 + Fe . \qquad (2)$$

The salt constituent UF_4 can give the reaction:

$$2UF_4 + Cr \rightleftharpoons CrF_2 + 2UF_3 . \qquad (3)$$

If salt containing UF_4 and a small amount of impurities is put into
a Hastelloy N system in which the salt circulates nonisothermally,
chromium will initially be removed from all parts of the system because
of reaction with both impurities and UF_4. The impurity reactions are
expected to go to completion fairly rapidly so that they will have an
effect only on the short-time corrosion results. On the other hand,
since the equilibrium constant for reaction (3) is a function of tempera-
ture, this reaction provides a means for the continuous transfer of

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[1]J. W. Koger, *Alloy compatibility with LiF-BeF₂ Salts Containing
ThF₄ and UF₄* ORNL/TM-4286 (December 1972).

[2]J. H. DeVan, *Effect of Alloying Additions on Corrosion Behavior of
Nickel-Molybdenum Alloys in Fused Fluoride Mixtures* M.S. Thesis, University
of Tennessee, August 1960.

chromium from the hotter sections of the system to the cooler sections. The amount of chromium, ΔM, removed from a unit area of surface can be shown to be:

$$\Delta M = BC_0 \sqrt{Dt} \qquad (4)$$

where B is a temperature-dependent constant, C_0 is the initial concentration of chromium in the alloy, and D is the diffusivity of chromium in the alloy. The limiting step for this mass transfer has been shown to be the diffusion of chromium in the metal when Hastelloy N is the alloy considered. If other strong fluoride formers — such as Ti, Nb, or Al — are present in the alloy, mass transfer of these elements would be expected to occur by the same mechanism as discussed above.

EXPERIMENTAL METHODS

Our corrosion studies have been carried out in five thermal-convection loops and one forced-circulation loop. Figures 1 and 2 show schematic drawings of these loops. These loops circulate salt around a system across which a temperature gradient is maintained. For most of these loops the temperature limits were maintained at 704 and 566°C (1300 and 1050°F), the proposed maximum and minimum temperatures for the fuel salt of an MSBR. Other important features of these loops are the removable corrosion specimens and the accesses to the salt to permit insertion of electrodes for controlled-potential voltammetry. Voltammetric measurements, which were made by members of the Analytical Chemistry Division, allowed us to make on-line determinations of the oxidation potential and corrosion product concentration of the salt. Detailed explanations of voltammetry are available elsewhere.[3,4] The operating conditions for each of the loops are described in Table 1.

[3]M. W. Rosenthal, P. N. Haubenreich, P. B. Briggs, Comps., *The Development Status of Molten Salt Breeder Reactors.*, ORNL-4812 (August 1972) pp. 153–57.

[4]J. R. Keiser, J. H. DeVan, and D. L. Manning, *The Corrosion Resistance of Type 316 Stainless Steel to Li_2BeF_4*, ORNL/TM-5782 (April 1977).

ORNL-DWG 68-3987R3

STANDPIPE

TWIN
BALL VALVES

SURGE
TANKS

CLAMSHELL
HEATERS

30 in.

INSULATION

CORROSION
SPECIMENS

SAMPLER

0   6
INCHES

FREEZE
VALVES

FLUSH
TANK

DUMP
TANK
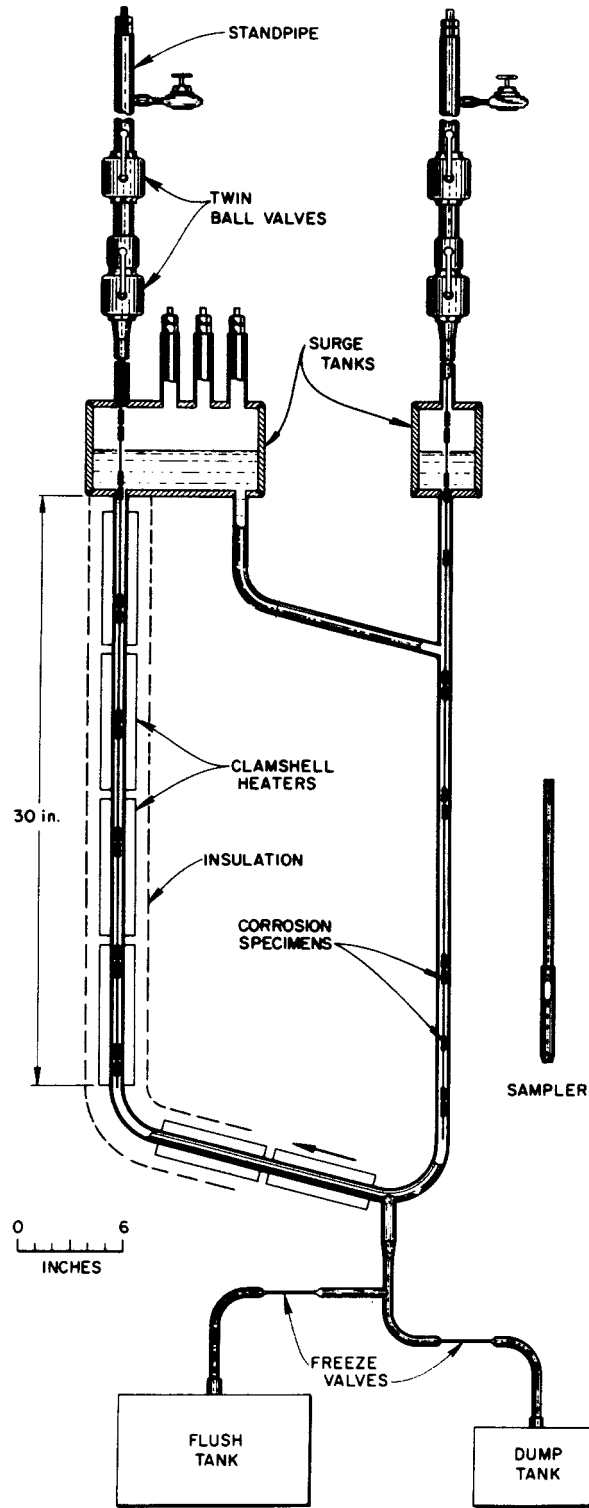
Fig. 1. Schematic of Thermal-Convection
Loop. Scale is 0.15 m. Height shown is 0.76 m.

FREEZE VALVE
(TYPICAL)

AIR

CORROSION SPECIMENS

HEATER LUGS (TYPICAL)

635°C
(1175°F)

RESISTANCE HEATED SECTION NO.1

THERMOCOUPLE
WELL

DRAIN AND FILL LINE

½-in.OD x 0.042-in.WALL
HASTELLOY N

BALL VALVE

AUXILIARY TANK

HEATER LUGS (TYPICAL)

RESISTANCE HEATED SECTION NO. 2

566°C
(1050°F)

COOLER NO. 1

ALPHA
SALT PUMP

FLOW RATE = ~4 gpm
VELOCITY = ~10 fps IN ½-in. TUBING
REYNOLDS NO. = 6600 TO 14,000

CORROSION
SPECIMENS

FREEZE VALVES

DRAIN AND
FILL LINE

COOLER NO. 2

CORROSION
SPECIMENS

FILL AND DRAIN TANK

705°C (1300°F)

DRAIN AND FILL LINE
¼-in.OD x 0.035-in. WALL

THERMOCOUPLE WELL

5
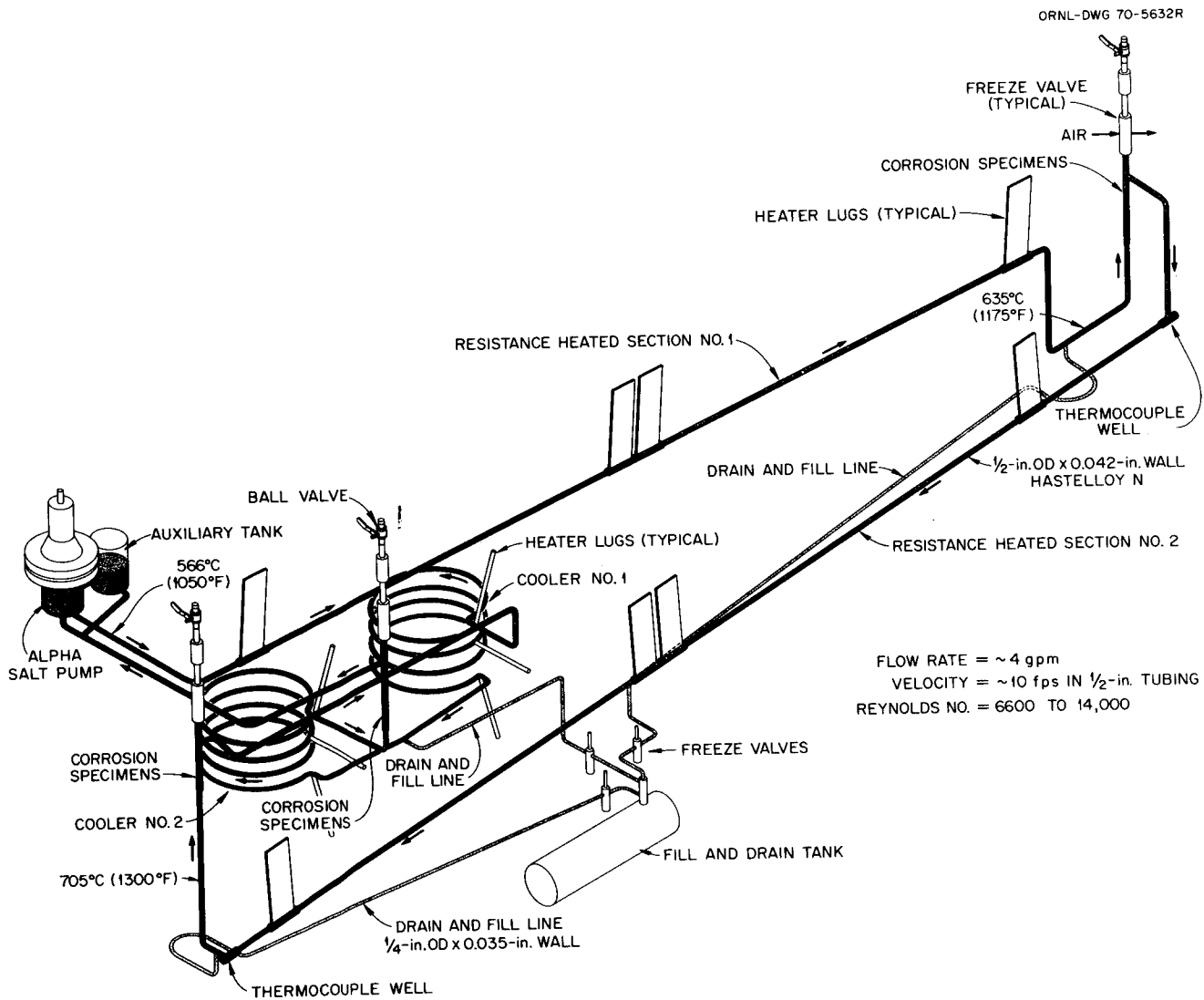
Fig. 2. Schematic of Molten-Salt Forced-Convection Corrosion Loop MSR-FCL-2 b.
Flow rate ≈ 0.25 liter/sec; velocity ≈ 3 m/sec in 13-mm tubing; loop tubing is
13-mm OD by 1.07-mm wall; drain and fill line is 6.4-mm OD by 0.9-mm wall.

Table 1. Loop Operating Conditions

Loop	Loop Material	Specimen Material	Salt	Operating Temperature, °C		Purpose
				Max	Min	
Thermal-Convection Loops						
21A	Hastelloy N	Hastelloy N, 1%-Nb-mod Hastelloy N	MSBR Fuel	704	566	1. Analytic method development 2. Base-line corrosion data
23	Inconel 601	Inconel 601	MSBR Fuel	704	566	1. Measure corrosion rate of high-Cr alloy (Inconel 601)
		Graphite	MSBR Fuel	677	550	2. Investigate Graphite UF_3 Reaction
31	Type 316 Stainless Steel	Type 316 Stainless Steel	Li_2BeF_4	649	493	1. Measure corrosion rate of type 316 stainless steel in potential coolant salt 2. Investigate effect of Be addition to salt
24	Hastelloy N	7%-Cr-mod Hastelloy N, 12%-Cr-mod Hastelloy N, 3.4%-Nb-mod Hastelloy N	MSBR Fuel	704	566	1. Measure corrosion rate of modified Hastelloy N alloys
18C	Hastelloy N	10%-Cr-mod Hastelloy N, 15%-Cr-mod Hastelloy N	MSBR Fuel	704	566	1. Measure corrosion rate of modified Hastelloy N alloys
Forced-Circulation Loop						
FCL-2B	Hastelloy N	Hastelloy N, 1%-Nb-mod Hastelloy N	MSBR Fuel	704	566	1. Base-line corrosion data in high-velocity salt

EXPERIMENTAL RESULTS

Thermal-Convection Loop 21A

Hastelloy N loop NCL 21A was the first loop to be put into operation
when the Molten-Salt Reactor Program was resumed in 1974. As such, the
loop was used to obtain base-line corrosion data for Hastelloy N and to
provide a test bed for voltammetry measurements of MSBR fuel salt. The
voltammetry results showed that the oxidation potential as reflected
by the U(IV)/U(III) ratio remained quite high throughout the 17.5 months
of operation. In fact, with a U(IV)/U(III) ratio of about 10^4 loop 21A
contained the most oxidizing salt of all the loop experiments.

The first specimens used in this loop were made of Hastelloy N
and were removed for examination about every 2500 hr. Figure 3 shows
the weight change as a function of the exposure time for these specimens
for up to 10,000 hr in salt. From this figure the rate of the weight
change clearly decreased with time. Figure 4 shows that the change
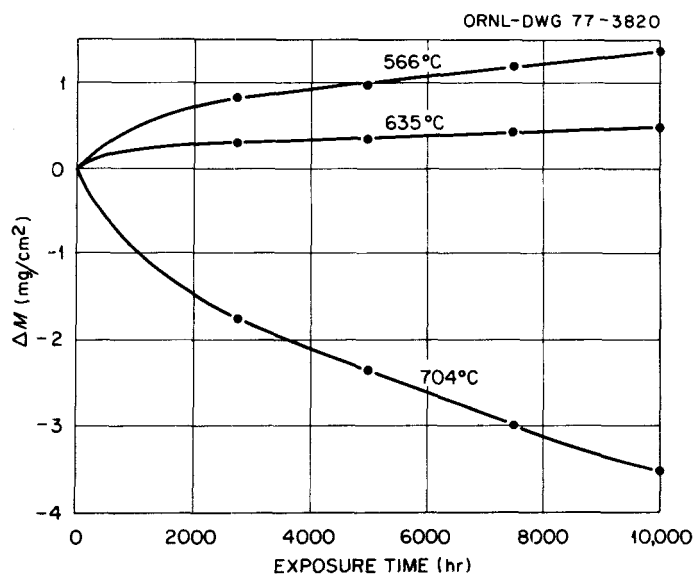in weight varies as the square root of time, as predicted from Eq. (4).

Fig. 3. Weight Change vs Exposure Time for Hastelloy N Specimens
Exposed to MSBR Fuel Salt at 566, 635, and 704°C in Thermal-Convection
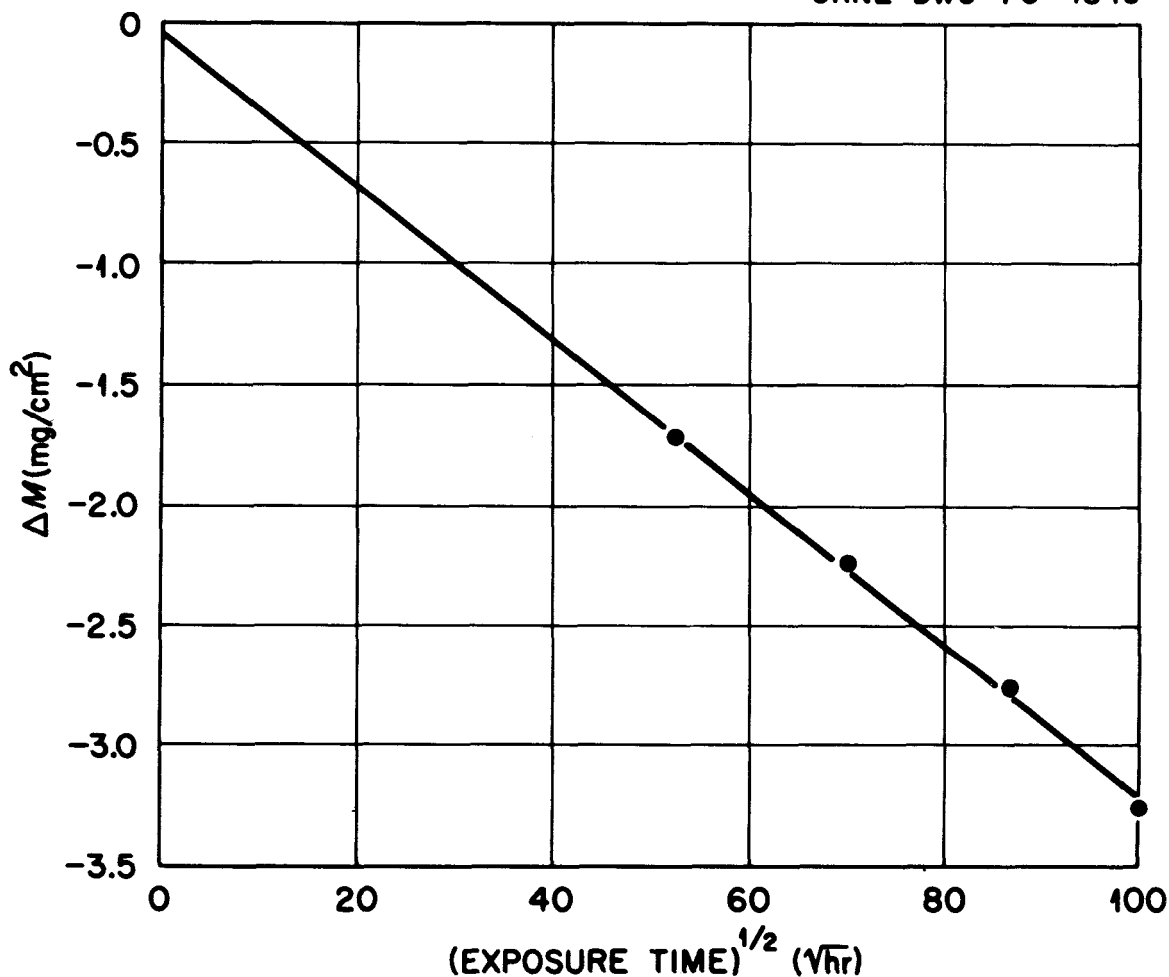Loop 21A.

ORNL-DWG 76-4843

Fig. 4. Weight Change vs Square Root of Exposure Time for Hastelloy N Specimen Exposed to MSBR Fuel Salt at 690°C in Thermal-Convection Loop 21A.

Specimens that had been exposed for 7500 hr in the hottest and coldest parts of the loop were examined metallographically. As is evident in Fig. 5 the pitting on the higher temperature specimen was limited to about 5 μm, indicating that the effect of the salt was relatively mild.

Following the 10,000-hr exposure of standard Hastelloy N, loop 21A was used to test specimens of 1%-Nb-modified Hastelloy N (experimental heat 522). Only a short exposure was achieved with these specimens before a power supply malfunction terminated the experiment, but the short-time corrosion results (Table 2) compare very favorably with results for standard Hastelloy N.
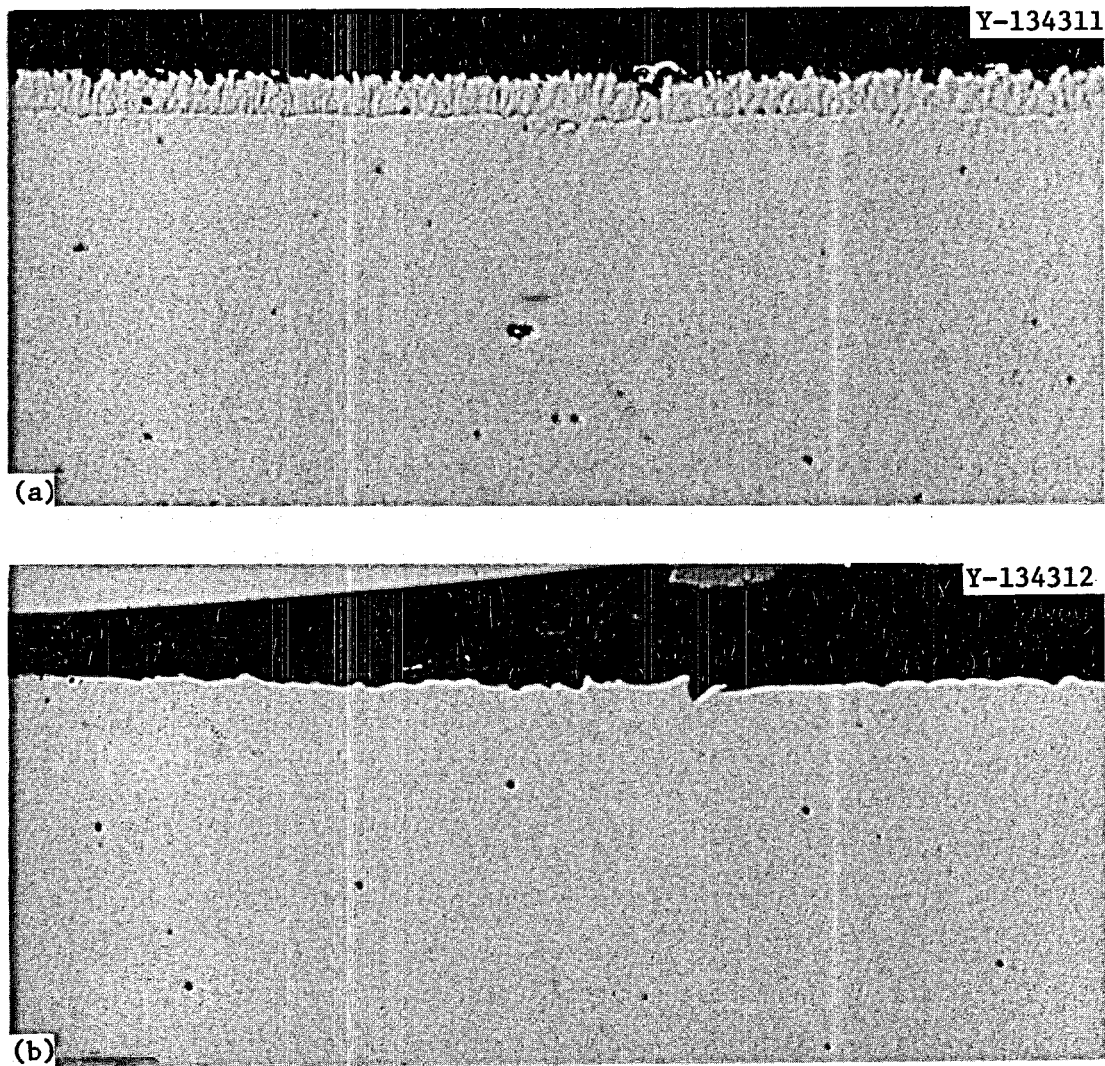
Fig. 5. Hastelloy N Exposed to MSBR Fuel Salt for 7500 hr at (a) 704 and (b) 566°C. 500×.

Table 2. Hastelloy N Corrosion Rate Measurements from Loop 21A

Alloy	Total Exposure (hr)	Corrosion Rate, mg cm^{-2} year^{-1}, at Each Exposure Temperature		
		566°C	635°C	704°C
Standard	10,009	+1.17	+0.39	−3.09
1% Nb Modified	1,004	0.0	−0.25	−3.30

Thermal-Convection Loop 23

The observation that the high-chromium alloy Inconel 601 (23 wt % Cr) resisted intergranular attack by tellurium led to the construction of a thermal-convection loop to determine how severe the corrosion by MSBR fuel salt would be. After the new loop was filled with salt, voltammetric techniques were used to follow the change in the U(IV)/U(III) ratio as an indication of the extent of the initial reaction between chromium and UF_4. The U(IV)/U(III) ratio decreased very rapidly, dropping to about 40 within a few days, meaning that considerable reaction was probably occurring between the salt and this Inconel 601 loop. Inconel 601 specimens exposed 721 hr all showed a weight loss, and that shown by the hottest specimen was very large (>30 mg cm^{-2} $year^{-1}$). Furthermore, the material lost by the hottest specimens was not removed uniformly from the surface, but resulted in the formation of the porous surface structure shown in Fig. 6. As shown in Fig. 7, electron microprobe examination of this specimen showed high thorium concentration in the pores. Since the only known source of thorium was the ThF_4 contained in the salt, the salt likely penetrated the pores. Continuous line scans made with the microprobe for the elements Ni, Cr, and Th, shown in Fig. 8, clearly show the depletion of chromium near the surface. These results provide further evidence of the presence of thorium in the pores. Diffusion calculations provide another piece of evidence indicating that salt must have penetrated the pores. Based on the bulk chromium concentration of 23 wt %, microprobe measurements[5] determined a chromium concentration of 6.6 wt % at the surface of the specimen in Fig. 6. From these concentrations, a depletion depth of 80 μm taken from Fig. 8, and diffusion values taken from Evans, Koger, and DeVan,[6] we calculate that the exposure time would have to be nearly 1000 times greater to attain this concentration profile as a result of bulk diffusion alone at 704°C. To obtain this

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[5]R. S. Crouse, ORNL, Private Communication, July 1975.

[6]R. B. Evans III, J. W. Koger, and J. H. DeVan, *Corrosion in Polythermal Loop Systems II. A Solid State Diffusion Mechanism With and Without Liquid Film Effects*, ORNL-4575, Vol. 2 (June 1971).
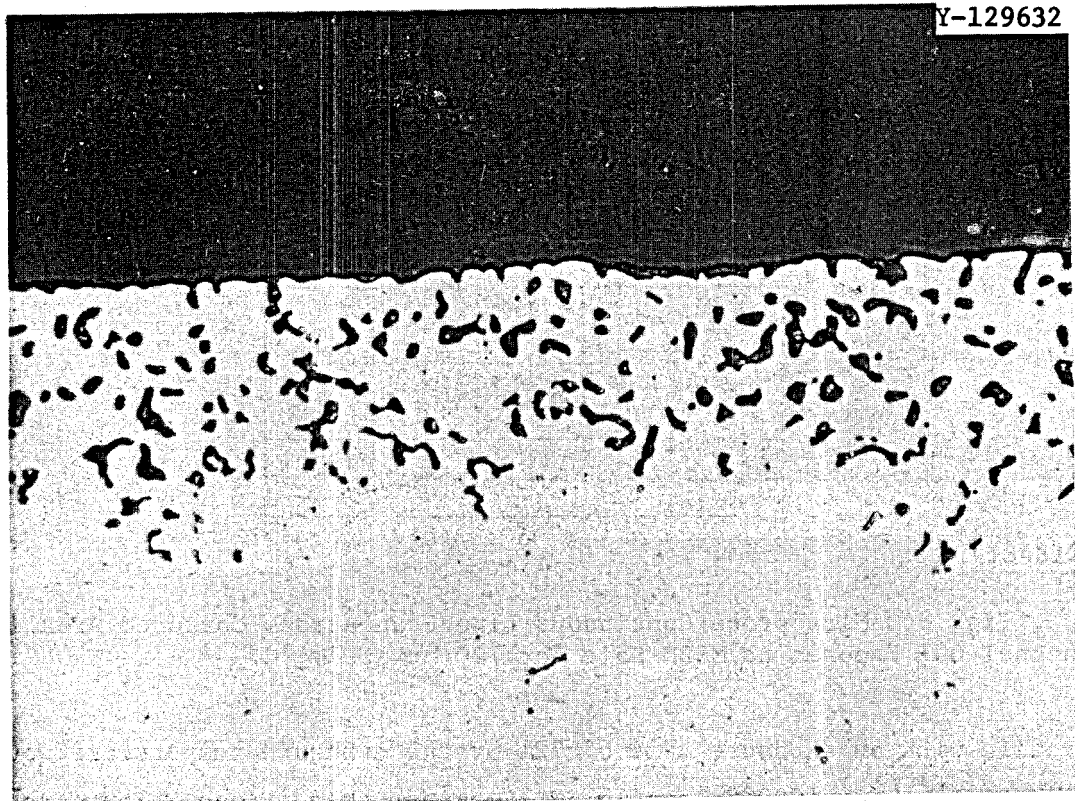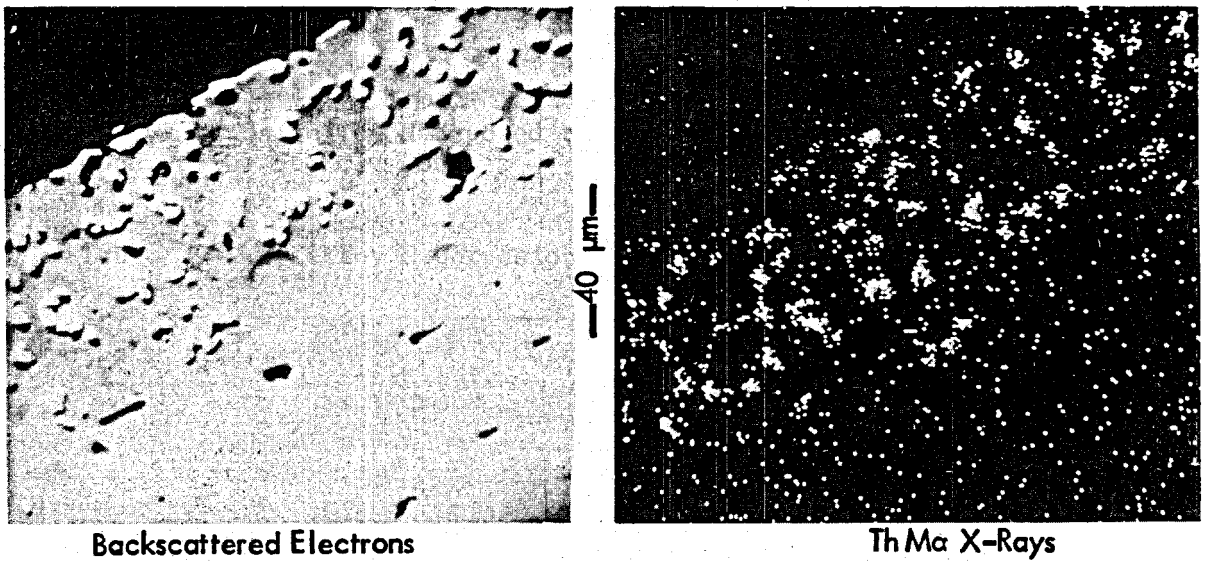
Fig. 6. Inconel 601 Exposed to MSBR Fuel Salt for 721 hr at 704°C. 500×.

Backscattered Electrons   Th Mα X-Rays

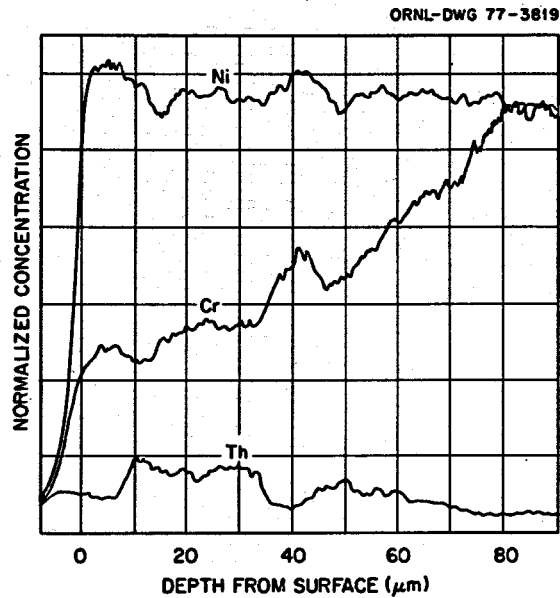Fig. 7. Electron-Beam Scanning Images of Inconel 601 Exposed to MSBR Salts for 720 hr at 704°C.

Fig. 8. Microprobe Continuous Line Scan Across Corroded Area in Inconel 601 Exposed to MSBR Salt for 720 hr at 704°C.

profile with an exposure time of 721 hr the exposure temperature would have to be about 1000°C, nearly 300°C higher than it was. Thus, to establish the gradient that was observed, salt was most likely present in the pores to provide a short-circuit path for removal of the chromium. Examination of a specimen from the coldest part of the loop revealed surface deposits, shown in Fig. 9, which were identified by microprobe analysis as chromium. The conclusion from this test is that Inconel 601 would be unsuitable for use in a molten-salt breeder reactor.
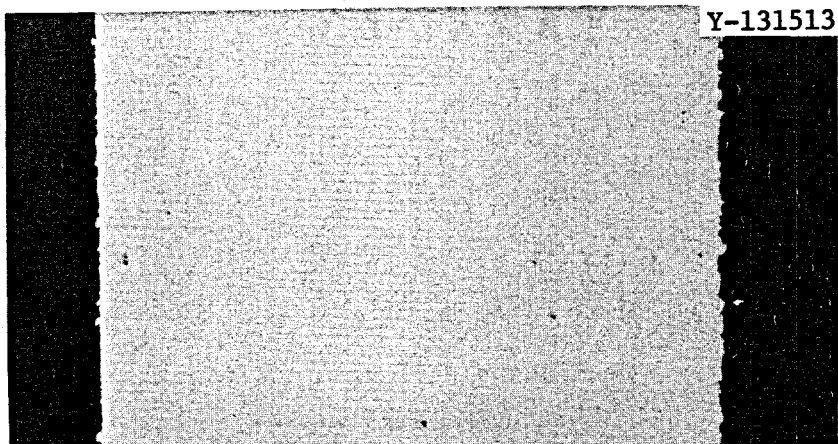

Fig. 9. Inconel 601 Exposed to MSBR Fuel Salt for 721 hr at 570°C. 500×.

It is expected that the lower limit for the U(IV)/U(III) ratio
in an MSBR will be determined by the conditions under which the reaction

$$4UF_3 + 2C \rightleftharpoons 3UF_4 + UC_2 \qquad (5)$$

proceeds to the right. Because the U(IV)/U(III) ratio of the salt
in loop 23 had decreased to less than 6, we decided to try to reproduce
the results of Toth and Gilpatrick,[7] shown in Fig. 10, which predict
that UC_2 should be stable at the lowest temperatures that could be
maintained in this loop, 545—550°C. However, graphite specimens ex-
posed to the salt for 530 hr did not show any evidence of UC_2. Since
the specimens used were made of pyrolytic graphite, the high density of
the material likely limited contact of the salt and graphite. The exper-
iment was repeated by exposing a less dense graphite for 530 hr,

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[7]L. M. Toth and L. O. Gilpatrick, *The Equilibrium of Dilute UF₃
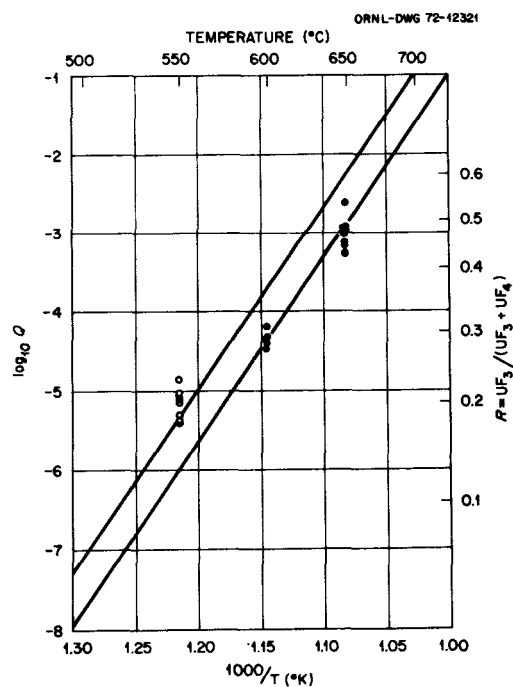Solutions Contained in Graphite*, ORNL/TM-4056 (December 1972).

Fig. 10. Equilibrium Quotients, $Q = (UF_3)^4/(UF_4)^3$, Versus Temperature
for $UC_2 + 3UF_4(d) \rightleftharpoons 4UF_3(d) + 2C$ in the Solvent $LiF-BeF_2-ThF_4$
(72-16-12 mole %).

then checking the graphite surface for the presence of a new phase by
x-ray diffraction. A new phase was found, and it was tentatively identi-
fied by O. B. Cavin as UO_2. If indeed this compound was UO_2, it probably
resulted from a uranium fluoride-water reaction, but it could have come
from the hydrolysis of UC_2 that had been formed by reaction (5). On
the basis of information from L. M. Toth[8] that nucleation of UC_2 under
our operating conditions could be very slow, a longer exposure was
undertaken. Two specimens of the less dense graphite were exposed for
about 3000 hr at a minimum temperature of 555°C in salt in which the
U(IV)U/(III) ratio had dropped to about 4. However, x-ray analysis of
these specimens showed no evidence of a phase other than the salt and
graphite. No further investigations were carried out because of the
termination of the program.

Thermal-Convection Loop 31

Thermal-convection loop 31 is constructed of type 316 stainless steel,
has type 316 stainless steel specimens, and has been used for corrosion
measurements with one of the alternative coolant salts, $LiF-BeF_2$
(66-34 mole %). For the first 1000 hr of operation this loop was used
to gather base-line corrosion data with as-received salt, which contained
a relatively high concentration of impurity FeF_2. As shown in Fig. 11,
fairly significant weight changes occurred in the specimens, especially
during the first 500 hr. Metallographic examination of specimens from
the hottest and coldest positions showed, respectively, pitting and
deposition, as is apparent in Fig. 12. Electron microprobe examination
of the deposits indicated they were predominately iron, and we expect
that the deposition occurred as a result of reaction (2). Bulk salt
analyses and voltammetric measurement of the FeF_2 and CrF_2 concentrations
of the salt during the first 1000 hr support this idea.

To learn if addition of a reductant to the salt would decrease
the impurity level and consequently lower the corrosion rate, beryllium
was added to the salt. New specimens were then inserted, and the corrosion
rate was measured for stainless steel in this "reducing" salt. As long
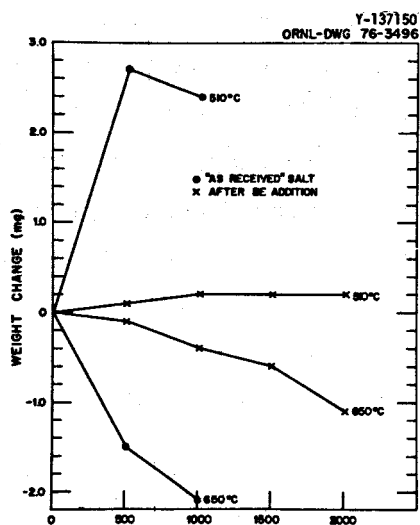
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[8]L. M. Toth, private communication.

15

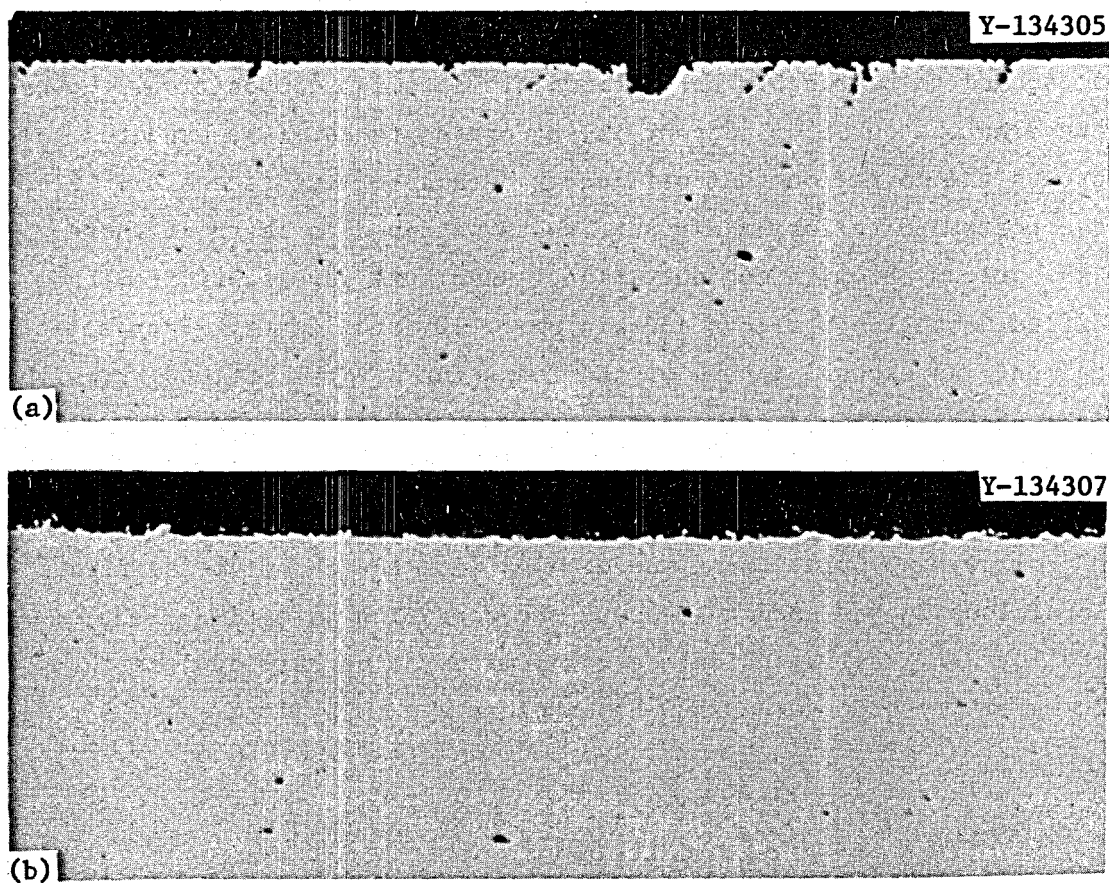Fig. 11. Weight Change vs Exposure Time for Type 316 Stainless Steel Exposed to LiF-BeF$_2$.

(a)

(b)

Fig. 12. Type 316 Stainless Steel Exposed to LiF-BeF$_2$ for 1000 hr at (a) 650 and (b) 510°C. 500×.

as the beryllium rod was in the salt, the corrosion rate was extremely low. This is shown in Fig. 11 for the first 500 hr after the addition of beryllium. After removal of the beryllium, the specimen in the hottest position showed a pattern of increasing weight loss as a function of time. This most probably occurred because, once the source of beryllium was removed, the species in the salt were no longer in equilibrium and the salt became progressively more oxidizing with increasing time, especially as moisture would leak into the system. Weight change results for specimens exposed to the "reducing" salt are also shown in Fig. 11.

Thermal-Convection Loops 18C and 24

Hastelloy N loops 18C and 24 were used to determine the effect of chromium concentration on the corrosion rate of Hastelloy N. Alloys such as stainless steels and Inconels with a relatively high chromium content had shown a resistance to grain boundary attack by tellurium that seemed to be roughly proportional to chromium composition. However, increasing the chromium content is, according to Eq. (4), expected to increase the corrosion that will occur because of mass transfer, From these tests and tellurium exposure tests we hoped to learn if there is an optimum chromium concentration. Specimens were fabricated from modified Hastelloy N alloys containing 7, 10, and 12% Cr. Each set of specimens was exposed in one of the loops for a total of 1000 hr, with weight change measured after 500 and 1000 hr. Within experimental error the weight change results (Fig. 13) show the dependence on initial chromium concentration that would be expected according to Eq. (4).

Following completion of the studies with the chromium-modified alloys, loop 24 was used to measure the corrosion rate of 3.4%-Nb-modified Hastelloy N. Results indicate a maximum corrosion rate of less than 2.20 mg cm^{-2} $year^{-1}$ for a 1500-hr exposure.
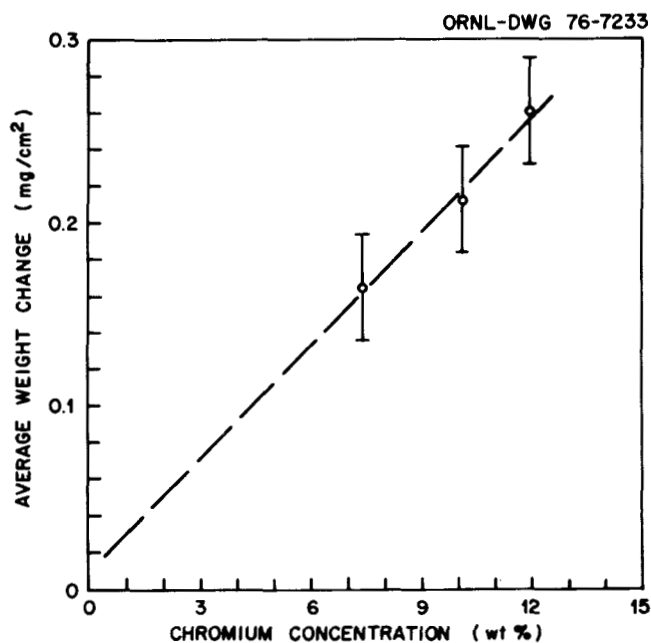
Fig. 13. Effect of Chromium Concentration in Modified Hastelloy N on Weight Change After 1000 hr in MSBR Fuel Salt.

Forced-Circulation Loop FCL-2B

Forced-circulation loop FCL-2B was used initially for fuel salt chemistry investigations. The first fuel salt corrosion investigations were made with standard Hastelloy N in salt with a U(IV)/U(III) ratio of about 100. Thus, the most significant differences between this loop and thermal-convection loop 21A were the oxidation potential of the salt [$\sim 10^2$ vs $\sim 10^4$ in terms of U(IV)/U(III) ratio] and the velocity of the salt 2.5—5 m/sec vs 1 m/min). If Eq. (4) and the ideas that led to its development are correct, we would expect that (1) the only effect of the low U(IV)/U(III) ratio in FCL-2B would probably be a reduction in the initial corrosion compared with NCL 21A, and (2) the high salt velocity would have no effect on mass transfer of chromium since the limiting factor is the diffusion of chromium to the surface of the alloy, not transfer of the chromium from the hot to the cold parts of the system. The results of this study, shown in Table 3, indicate a very low corrosion rate for the 4000-hr exposure. This test was interrupted several times for repairs on the loop and for heat transfer measurements, but that did not seem to have a detrimental effect on the results.

A 4000-hr corrosion test of Hastelloy N in fuel salt with a
U(IV)/U(III) ratio of 1000 was planned. Because of the decision to
terminate the Molten-Salt Reactor Program, we decided the remaining
operating time for this loop could be best spent measuring the corrosion
rate of 1%-Nb-modified Hastelloy N, an alloy that had shown good resistance
to tellurium attack. Accordingly, a set of such specimens was prepared
and inserted into the loop. Because of loop operating difficulties some
of the weight changes measured after 500 and 1000 hr were of questionable
value. Measurements made after 1500- and 2200-hr exposure were of good
quality and are summarized in Table 3 along with some of the 500- and
1000-hr results. Included in this table for comparison are the results
for the standard Hastelloy N specimens exposed for 4309 hr in this same
salt and loop. It should be noted that the niobium-modified specimens
gained more weight than they lost. This is most probably due to mass
transfer of iron from the standard Hastelloy N tubing, which contains
about 5 wt % Fe, to the modified alloy, which has essentially no iron.
Taking into account the value at which the corrosion rate of the modified
Hastelloy N seems to be leveling out, we expect that the corrosion resis-
tance of the 1%-Nb-modified Hastelloy N is at least as good as that of
standard Hastelloy N.

Table 3. Corrosion Rate Measurement for Hastelloy N
Specimens Exposed in FCL-2B

Alloy	Exposure	Corrosion Rate, mg cm^{-2} $year^{-1}$, at Each Exposure Temperature		
		566°C	635°C	704°C
Standard	4309 hr	+0.02	−0.20	−2.31
1%-Nb-Modified	first 580 hr			−0.52
	next 497 hr			−0.31
	next 496 hr	+1.69	+2.31	−0.38
	next 669 hr	−0.46	+0.91	−0.28

CONCLUSIONS

The following conclusions can be drawn from this work.

1. Voltammetry provides a very convenient means for on-line measurement of the oxidation potential and impurity concentration of the salt.

2. Inconel 601 would probably not have sufficient corrosion resistance to be acceptable for use as a containment vessel material.

3. No evidence of the formation of uranium carbide was found in studies of the reaction of graphite with very reducing salt.

4. Weight loss of Hastelloy N specimens occurred as a linear function of the square root of the exposure time indicating diffusion-controlled corrosion.

5. Type 316 stainless steel has a high initial corrosion rate in as-received Li_2BeF_4, but has a very low corrosion rate when beryllium is added to the salt.

6. Alloys of Hastelloy N modified by the addition of chromium showed weight losses proportional to the chromium concentration.

7. Limited results indicate that the corrosion rates of 1- and 3.4%-Nb-modified Hastelloy N are at least as good as that of standard Hastelloy N.

ACKNOWLEDGMENTS

INTERNAL DISTRIBUTION

1-2.	Central Research Library	51.	A. D. Kelmers
3.	Document Reference Section	52.	R. E. MacPherson
4-13.	Laboratory Records Department	53.	G. Mamantov
14.	Laboratory Records, ORNL RC	54.	D. L. Manning
15.	ORNL Patent Office	55.	C. L. Matthews
16.	C. F. Baes	56.	L. Maya
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28.	J. H. DeVan	68.	L. M. Toth
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