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Status of Tellurium-Hastelloy N Studies in Molten Fluoride Salts

J. R. Keiser

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STATUS OF TELLURIUM-HASTELLOY N STUDIES IN MOLTEN FLUORIDE SALTS

J. R. Keiser

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CONTENTS

	Page
ABSTRACT	1
INTRODUCTION	1
TELLURIUM EXPERIMENTAL POT 1	2
CHROMIUM TELLURIDE SOLUBILITY EXPERIMENT	4
Ni_3Te_2 CAPSULE TEST	10
TELLURIUM SCREENING TEST	13
CHROMIUM-TELLURIUM-URANIUM INTERACTION EXPERIMENT	13
SUMMARY	22
ACKNOWLEDGMENTS	22
REFERENCES	23

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J. R. Keiser

ABSTRACT

Tellurium, which is a fission product in nuclear reactor fuels, can embrittle the surface grain boundaries of nickel-base structural materials. This report summarizes results of an experimental investigation conducted to understand the mechanism and to develop a means of controlling this embrittlement in the alloy Hastelloy N. The addition of a chromium telluride to salt can be used to provide small partial pressures of tellurium simulating a reactor environment where tellurium appears as a fission product. The intergranular embrittlement produced in Hastelloy N when exposed to this chromium telluride-salt mixture can be reduced by adding niobium to the Hastelloy N or by controlling the oxidation potential of the salt in the reducing range.

INTRODUCTION

Hastelloy N composed of 7% Cr, 16% Mo, balance nickel, was used to contain the $LiF-BeF_2-ZrF_4-UF_4$ fuel salt in the Molten Salt Reactor Experiment (MSRE). Tensile testing of Hastelloy N surveillance specimens, which had been exposed to fuel salt in the MSRE, produced cracks in the grain boundaries connecting to the salt-exposed surfaces of the specimens. Surface chemical analyses of the affected grain boundaries indicated the presence of the fission product tellurium.[1,2] Because the depth of cracking observed in the MSRE would not be acceptable when extrapolated to the 30-year design life of a Molten-Salt Breeder Reactor (MSBR), work began to provide for the MSBR primary containment vessel a material that would resist grain boundary attack by tellurium. In-reactor experiments, identified as TeGen capsules, were used to evaluate the embrittlement resistance of various alloys, but these experiments were time consuming

and expensive, and the number of alloys that could be tested at any one time was limited.

To evaluate many alloys and modifications for their resistance to tellurium attack, a test was sought to simulate the appearance of tellurium as a fission product in reactor fuel salt. A total of five experiments have been conducted to learn more about the behavior of various tellurium compounds in salt and to determine the effects of these telluride-salt mixtures on potential containment vessel materials.

The tellurium effect was measured in terms of the number and depth of cracks produced in a tensile specimen that had been strained to failure at room temperature after exposure in a molten salt that contained the telluride. Straining was used to open visible cracks along embrittled grain boundaries to facilitate counting, but it also caused a few other cracks, which were unrelated to the presence of tellurium, to form. These other cracks were factored into our analysis.

The results of the tellurium-Hastelloy N-molten fluoride salt studies follow in chronological order. The last experiments were not completed because the MSBR program was terminated.

TELLURIUM EXPERIMENTAL POT 1

Our first tellurium experiment (pot 1) was in the form of a static pot built to evaluate the use of lithium telluride as a means for adding tellurium to salt. This pot (Fig. 1) allows tellurium to be added periodically in the form of salt pellets containing a measured amount of lithium telluride. Three viewing ports permit observation of the pellets after their addition to the salt. Electrodes for voltammetric measurements of the salt are inserted through Teflon seals and are used to detect and measure the concentration of certain species in the salt. About 600 ml of the salt $LiF-BeF_2-ThF_4$ (72-16-12 mol %) was used, and its temperature was maintained at 650°C.

In the first experiment, tellurium was added as the lithium telluride, Li_2Te, which was prepared by the ORNL Chemistry Division. Initially, two pellets, containing a total of 70 mg of Li_2Te, were added to 600 ml of salt. Voltammetric examination of the salt by D. L. Manning of the

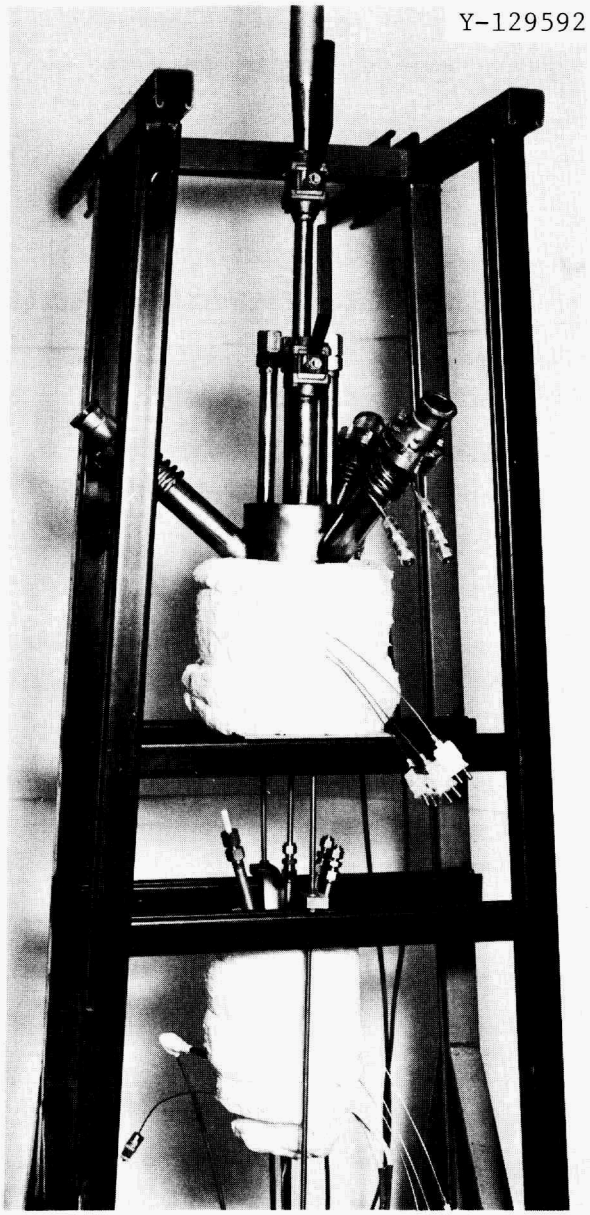

Fig. 1. Tellurium Experimental Pot 1 Showing the Three Viewing Ports and Accesses for Electrochemical Probes.

Analytical Chemistry Division gave no indication of the presence of tellurium. Subsequently, three more Li_2Te pellets were added, and the salt was sampled for chemical analysis. Three additions of CrF_2 totalling 1.82 g were then made, followed by the addition of three more Li_2Te pellets. A final addition consisting of 0.2 g of BeO was made.

Results are summarized as follows:

1. The Li_2Te pellets did not melt and disappear immediately; some evidence of the pellets remained on the salt surface for the duration of the experiment.

2. No electrochemical evidence of a soluble tellurium species was detected.

3. Following the Li_2Te additions, visibility through the view ports was limited by a bluish-grey deposit that was subsequently identified as predominantly tellurium.

4. Chemical analysis of the salt sample taken after the addition of five Li_2Te pellets showed that the tellurium content was less than 5 ppm. The conclusion was that use of Li_2Te is not feasible for adding tellurium to MSBR fuel salt.

A second experiment was conducted in the same static pot at 650°C using the reportedly more soluble lithium telluride, $LiTe_3$. After the pot had been drained and cleaned, fresh salt was added, and three pellets containing a total of about 0.1 g of $LiTe_3$ were inserted. During the following three weeks, electrochemical examinations of the salt by D. L. Manning revealed no indication of a soluble tellurium species. Since other experiments had shown that metal specimens could be embrittled by tellurium even when tellurium levels in the salt were below chemical detection limits, a Hastelloy N tensile specimen was inserted into the salt and three additional $LiTe_3$ salt pellets were added. After about 650 hr the specimen was removed from the salt, tensile-tested to failure, and examined metallographically. Intergranular cracking, probably due to tellurium, was found, indicating that some mechanism exists for transport of tellurium or $LiTe_3$ through the salt. Because concurrent experiments had identified simpler methods of adding tellurium to salt, no further work was carried out with either of the lithium tellurides.

CHROMIUM TELLURIDE SOLUBILITY EXPERIMENT

The addition of a soluble chromium telluride — either Cr_2Te_3, Cr_3Te_4, or Cr_5Te_6 — represents another method for adding tellurium to molten MSBR fuel salt. If an excess of chromium telluride is maintained, the chemical activity of tellurium in solution in the salt will be

determined if the temperature and the salt composition are not changed. To determine whether there is a temperature at which one of these chromium tellurides can provide a reasonable amount of tellurium in solution, we determined solubility of the chromium tellurides as a function of temperature.

A Hastelloy N pot was filled with about 500 ml of the salt $LiF-BeF_2-ThF_4$ (72-16-12 mol %), and the temperature of the pot was controlled at 700°C. After a sample of the salt was taken, Cr_3Te_4 was added and a small Hastelloy N sheet specimen was inserted into the salt. After 170 hr the specimen was removed and after 250 hr a salt sample was taken. The temperature was then lowered to 650°C, and a day later another salt sample was taken.

This sequence was then repeated at 600°C. Next, the salt temperature was raised to 700°C, Cr_2Te_3 was added, and another Hastelloy N specimen inserted. The specimen was removed and salt samples were taken under the same time-temperature conditions as discussed above.

The two Hastelloy N specimens were weighed and submitted for Auger electron spectroscopic (AES) examination. No weight changes were detected, but evidence of tellurium in the grain boundaries was found by AES. The results of the chemical analysis of the salt sample are shown in Table 1.

Table 1. Results of Chromium Telluride Solubility Measurements

Sampling temperature (°C)	Tellurium and Chromium Content of Salt Samples (ppm)		
	Background (no Te added)	After Cr_3Te_4 Addition	After Cr_2Te_3 Addition
700	Te <5 Cr 44	Te <5 Cr 75	Te <5 Cr 90
650		Te 15.1 Cr 105	Te 7.5 Cr 120
600		Te <5 Cr[a]	Te <5 Cr 88

[a]Insufficient sample.

Tellurium concentrations at 700°C were not as high as was expected, but some tellurium was in solution as demonstrated by the tellurium found on the grain boundaries of the specimens.

Following the solubility measurements, two tensile specimens were exposed to the salt-Cr_2Te_3 solution. Both specimens, one of standard Hastelloy N and one of 2.6% Nb–0.7% Ti-modified Hastelloy N, showed a weight increase after 500 hr exposure at 700°C. After room-temperature tensile testing, the standard Hastelloy N specimen had significantly more and deeper cracks than did the modified Hastelloy N specimen, as can be seen in Fig. 2 and Table 2. Also exposed to this salt-Cr_2Te_3 solution were eight specimens that had been specially prepared for examination by AES. Unfortunately, the annealing treatment used before test resulted in large grains, and the data obtained were not interpretable.

Because specimens exposed to the Cr_2Te_3-salt solution had a surface reaction layer, we decided to use Cr_3Te_4 for future experiments because of its lower tellurium activity. Accordingly, another pot was prepared, filled with salt, and doped with Cr_3Te_4. Tensile specimens of several modified Hastelloy N alloys with different combinations of niobium, titanium, and chromium were exposed to the salt-Cr_3Te_4 system at 700°C. Examination of these specimens after tensile testing gave results included in Table 2. These results indicate that niobium as an alloying agent reduces embrittlement of Hastelloy N, but neither chromium nor niobium with titanium exhibit as strong an effect.

Standard Hastelloy N specimens intended for Auger studies were also exposed in this salt-Cr_3Te_4 system to provide information about the rate of tellurium attack (Fig. 3). As part of the Auger examination of these specimens, the depth of tellurium penetration was measured as a function of exposure time.[3] These results are shown in Fig. 4.

For grain boundary diffusion where the diffusing species is soluble in the bulk, a 1/4 power dependence is predicted. If essentially no bulk solubility is assumed, a power dependence of 1/2 is expected. As seen from Fig. 4, these data fit a $t^{1/2}$ plot reasonably well. Because the exposure times considered were relatively short and because scatter in the measurements of depth was fairly large, the penetration rate calculated from these data has a rather large standard deviation.

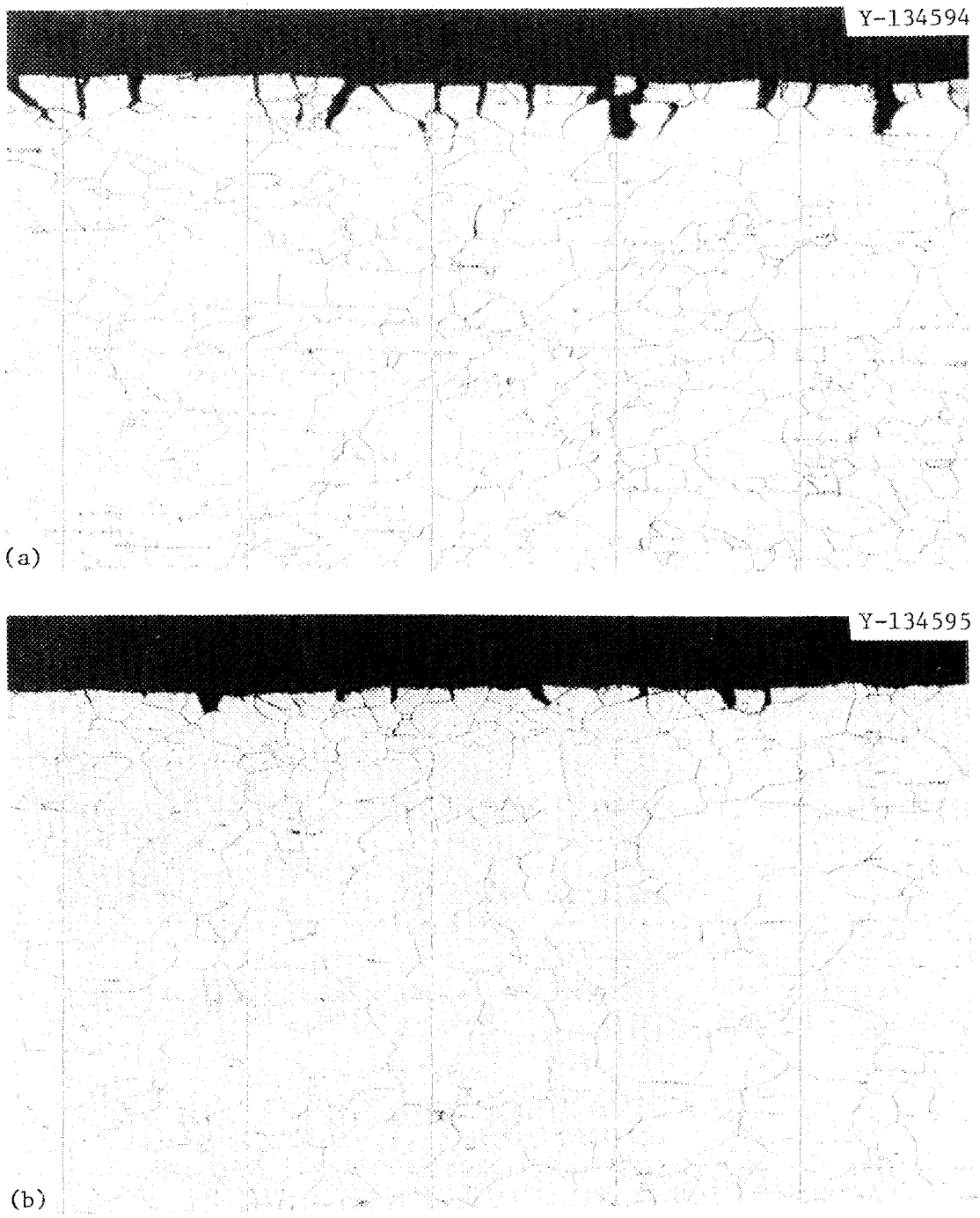

Fig. 2. Photomicrographs of (a) Standard Hastelloy N, and (b) 2.6% Nb–0.7% Ti-modified Hastelloy N Exposed to Salt plus Cr_2Te_3 for 500 hr at 700°C, Then Strained to Failure at Room Temperature. Etched with aqua regia. 100×.

Table 2. Intergranular Crack Behavior of Hastelloy N Specimens Exposed in Chromium Telluride Solubility Experiment

Alloy	Exposure[a] Time (hr)	Salt Additive	Crack[b] Density (m^{-1})	Average Crack Depth (μm)	Density Times Depth
Standard	504	Cr_2Te_3	1.46×10^4	42.5	0.6205
2.6% Nb, 0.7% Ti	504	Cr_2Te_3	0.71	33.1	0.2350
Standard	499	Cr_3Te_4	1.38	50.4	0.6955
2.6% Nb, 0.7% Ti	499	Cr_3Te_4	0.97	24.3	0.2357
1.0% Nb, 1.0% Ti	244	Cr_3Te_4	1.02	37.4	0.3815
1.0% Nb	244	Cr_3Te_4	0.17	30.5	0.0519
7% Cr	500	Cr_3Te_4	0.47	35.6	0.1673
10% Cr	500	Cr_3Te_4	0.28	41.3	0.1156
12% Cr	503	Cr_3Te_4	0.71	43.7	0.3103
15% Cr	503	Cr_3Te_4	0.57	48.9	0.2787

[a]All exposures were at 700°C.

[b]Measured number of cracks per meter of length along edge of a polished cross section of specimen.

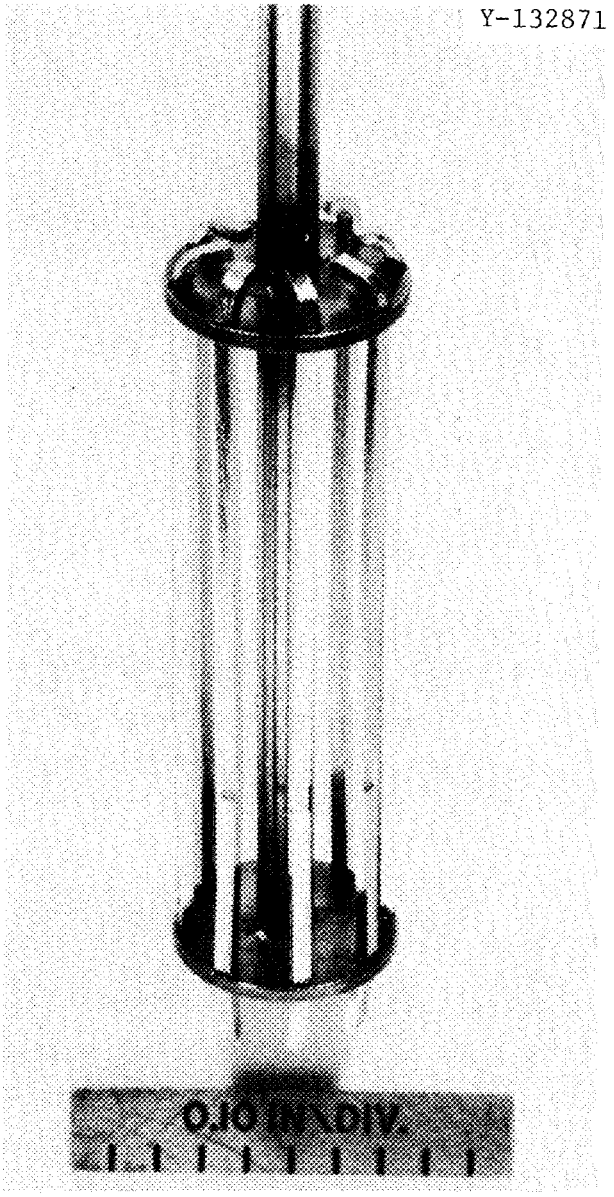

Fig. 3. Hastelloy N Tensile Specimens Designed for Auger Studies Mounted in Holder Used for Salt Exposures. Note the reduced section in the lower portion of the specimens.

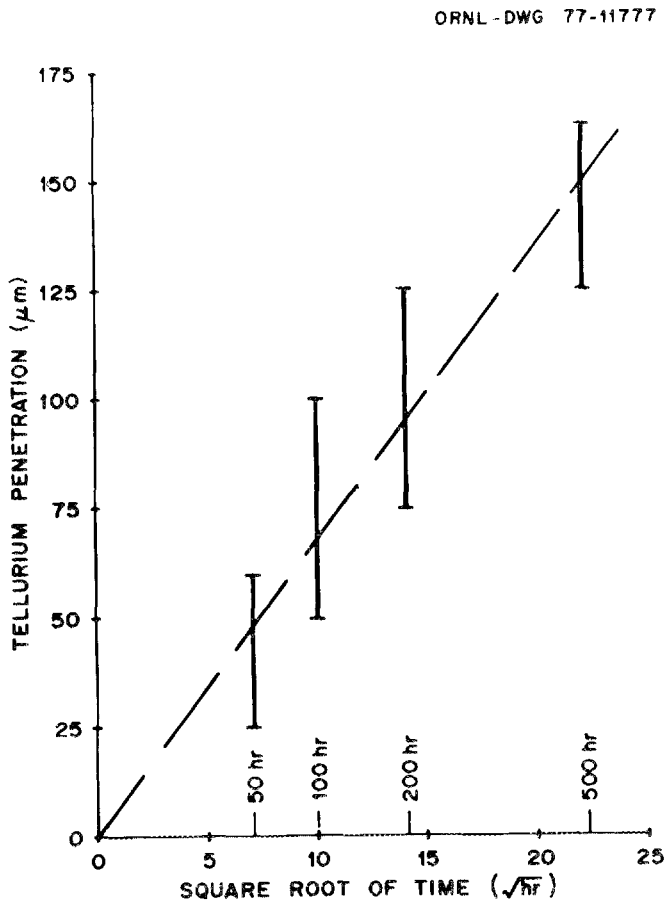

Fig. 4. Tellurium Penetration versus Time for Hastelloy N Exposed at 700°C to $LiF-BeF_2-ThF_4$ (72-16-12 mol %) Containing Cr_3Te_4. Data obtained by AES.

Ni_3Te_2 CAPSULE TEST

Brynestad[4] reported that a Hastelloy N specimen exposed to vapor above Ni_3Te_2 + Ni for 1000 hr at 700°C did not show intergranular cracking. This suggested that the Ni_3Te_2 + Ni system has a tellurium activity at which Hastelloy N is not attacked. To further evaluate this potentially significant result, a test was designed to expose Hastelloy N tensile specimens to salt containing Ni_3Te_2 + Ni. A capsule was built and filled with about 310 ml of $LiF-BeF_2-ThF_4$ salt. A mixture of 62 at. % Ni—38 at. % Te, which had been annealed at high temperature to promote formation of Ni_3Te_2 plus a small excess of nickel, was added to the capsule, and four Hastelloy N specimens were inserted. One specimen was removed after 1000 hr at 700°C, strained to failure, and examined

metallographically. Contrary to Brynestad's earlier results in vapor, extensive intergranular cracking was found after exposure in salt. An x-ray examination[5] of the Ni_3Te_2 + Ni mixture showed no evidence of unreacted tellurium, indicating that even the low tellurium activity associated with Ni_3Te_2 in this system causes intergranular attack of Hastelloy N. All three remaining specimens, which were exposed for longer times at 700°C, showed intergranular attack, and the results of metallographic examinations are given in Table 3.

Table 3. Intergranular Crack Behavior of Hastelloy N Exposed in Ni_3Te_2 Capsule Test at 700°C

Alloy	Exposure Time (hr)	Crack Density (m^{-1})	Average Crack Depth (μm)	Density Time Depth
Standard	1079	8700	19.7	0.1714
Standard	2297	7700	34.8	0.2680
Standard	4377	9400	36.1	0.3393
Standard	4377	4700	34.7	0.1631
1.1% Nb	2080	300	48.2	0.0145
2.0% Nb	2080	1300	36.2	0.0471
Standard	2976	7900	23.2	0.1833
1.1% Nb	2976	400	44.3	0.0177
2.0% Nb	2976	800	37.5	0.0300
4.4% Nb	2976	4000	29.1	0.1164

Because other experiments were indicating that alloying with 1 to 2% Nb lessens the tellurium embrittlement of Hastelloy N, four additional modified specimens were exposed to this Ni_3Te_2-salt mixture in the capsule described above. The specimens contained additions of 0, 1.1, 2.0, and 4.4% Nb and all were exposed for about 3000 hr at 700°C. After exposure, metallographic examination of the tensile tested specimens confirmed the effect of small additions of niobium on the tellurium embrittlement of Hastelloy N. Results are included in Table 3. Photomicrographs of these specimens are shown in Fig. 5.

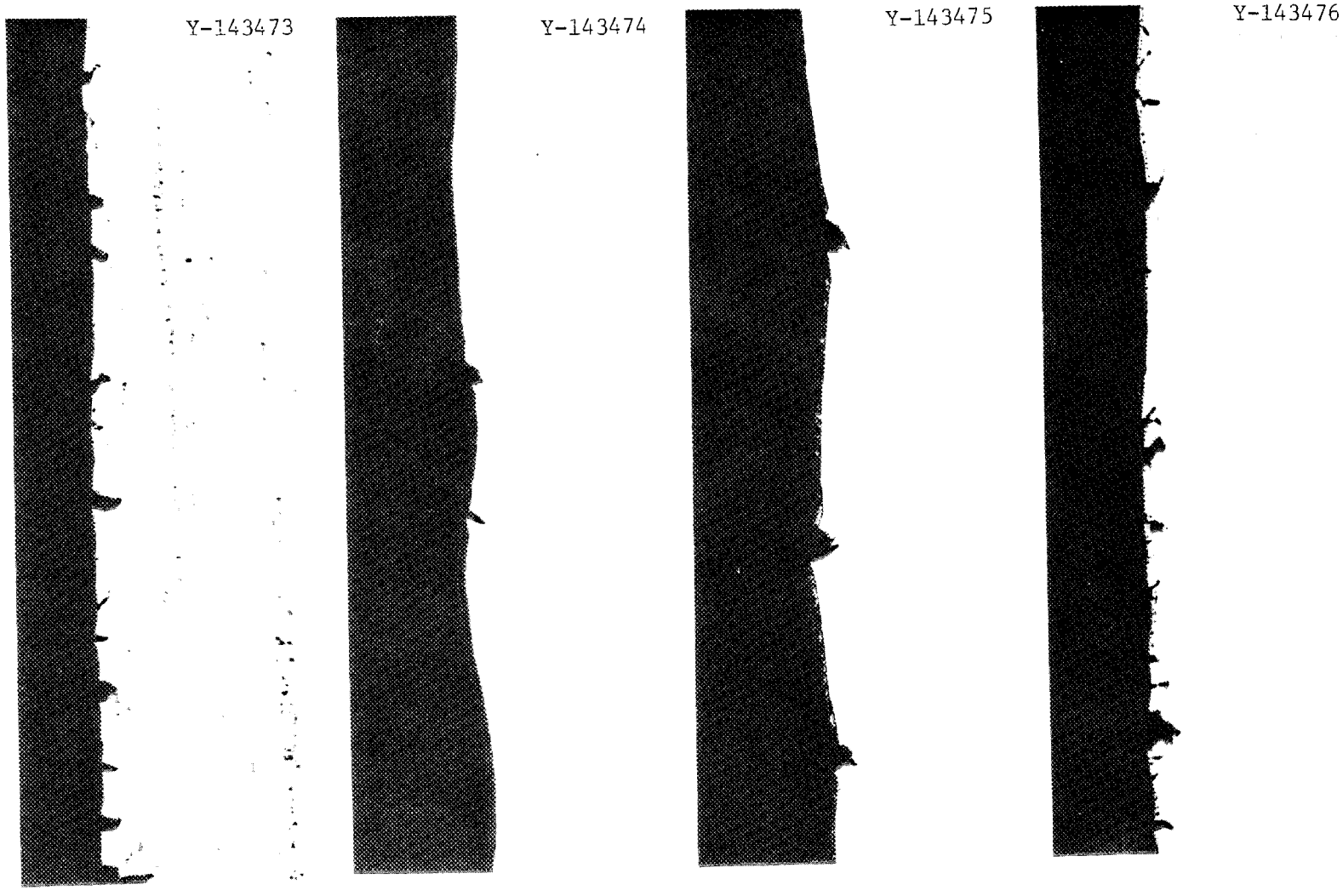

Fig. 5. Effect of Niobium on the Tellurium Embrittlement of Hastelloy N that Had Been Exposed About 3000 hr at 700°C to $LiF-BeF_2-ThF_4$ (72-16-12 mol %) Containing Ni_3Te_2 + Ni. 100×.

TELLURIUM SCREENING TEST

To compare the resistance of various alloys to tellurium attack, we constructed a large pot to permit simultaneous exposure of a large number of specimens to a salt-tellurium system. This pot is equipped with a stirring mechanism, electrochemical probes, and five accesses for insertion of specimens. Up to 100 specimens can be exposed to salt at one time (Figs. 6 and 7). The pot was filled with about 11 liters of $LiF-BeF_2-ThF_4$ salt, and a mixture of Cr_3Te_4 and Cr_5Te_6 was added. Specimens that had been selected from the more than 50 different available modified and standard Hastelloy N compositions were exposed to this salt-telluride mixture for times ranging from 250 to 5000 hr at 700°C. Standard Hastelloy N was cracked to about the same extent when exposed in this system as when exposed in previously discussed systems. All results of the various exposures are tabulated elsewhere.[6] The most notable result of these tests was the observation that the addition of 1 to 2 at. % Nb to Hastelloy N significantly reduced the cracking, as shown in Figs. 8 and 9. A more thorough discussion of these results will be given elsewhere.[6]

CHROMIUM-TELLURIUM-URANIUM INTERACTION EXPERIMENT

The research effort to solve the problem of tellurium intergranular attack of Hastelloy N was predominantly concerned with the development and testing of new alloys. The possibility of a chemical change in the salt that could alter the extent of attack by tellurium was also considered. It has been suggested[7] that since chromium ion activity can be controlled, within a certain range, by the U(IV)/U(III) ratio, it might be possible to control tellurium activity by "complexing" reactions with chromium or other salt constituents, for example,

$$2UF_3 + MF_2 + xTe \rightleftharpoons \text{"}MTe_x\text{"} + 2UF_4 \ ,$$

where M denotes chromium or another salt component.

Electrochemical studies reported by Manning and Mamantov[8] indicated that in a relatively reducing $LiF-BeF_2-ThF_4$ melt, tellurium could exist

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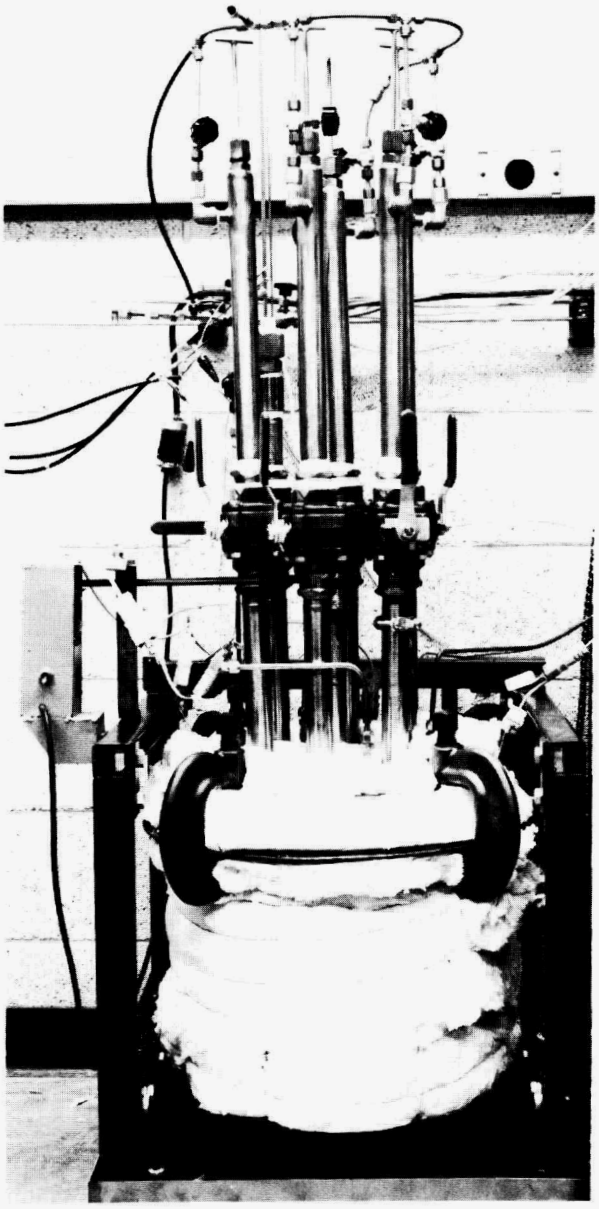

Fig. 6. Tellurium Screening Pot Used for Exposing Tensile Specimens to $LiF-BeF_2-ThF_4$ (72-16-12 mol %) Containing $CrTe_{1.266}$.

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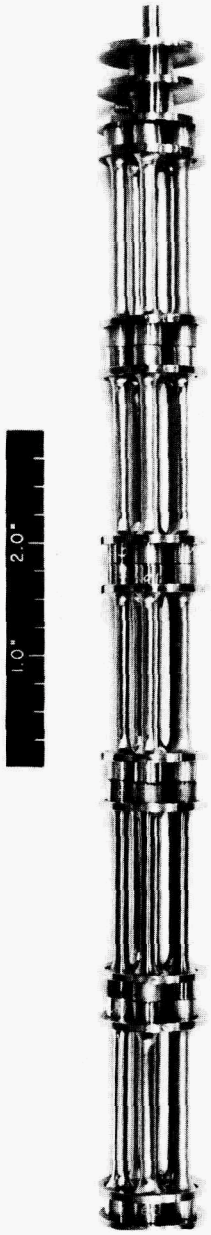


Fig. 7. Holder with 25 Tensile Specimens Prepared for Insertion in Tellurium Screening Pot. The five top specimens will be in the vapor space above the salt, while the other 20 specimens will be in the molten salt.

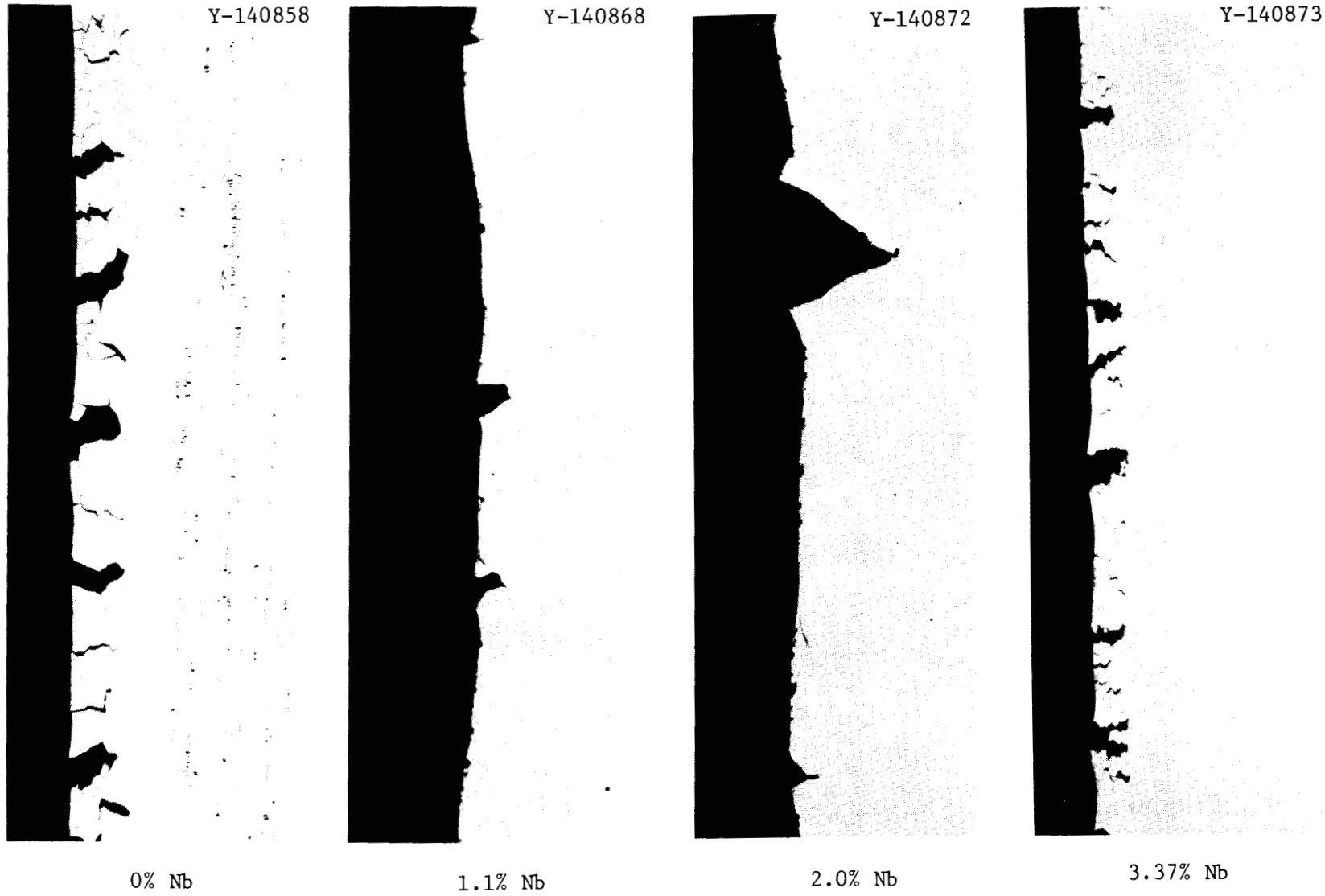


Fig. 8. Effect of Niobium Additions on the Embrittlement of Hastelloy N Exposed 1000 hr at 700°C to $LiF-BeF_2-ThF_4$ (72-16-12 mol %) Containing $CrTe_{1.266}$. 100×.

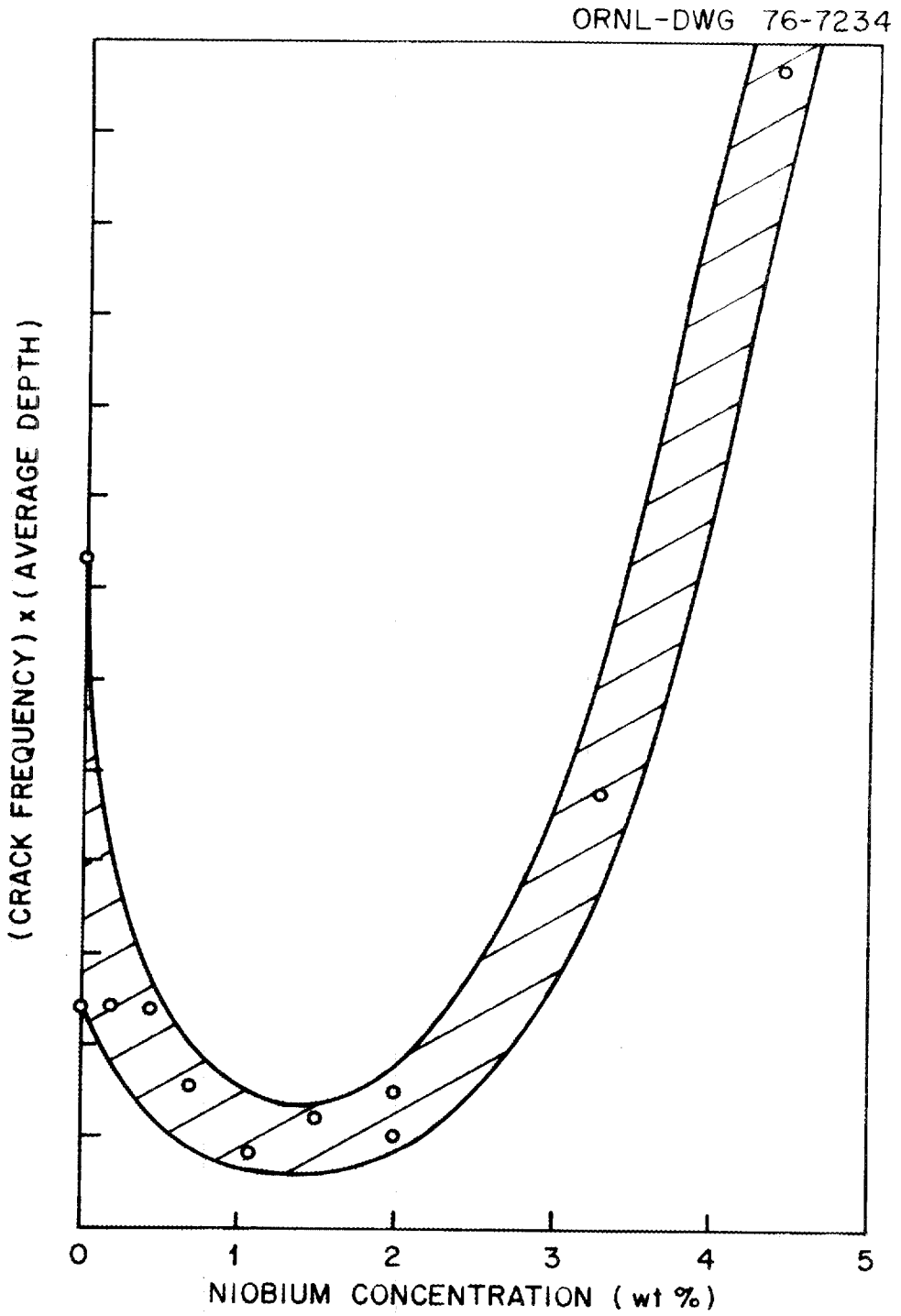


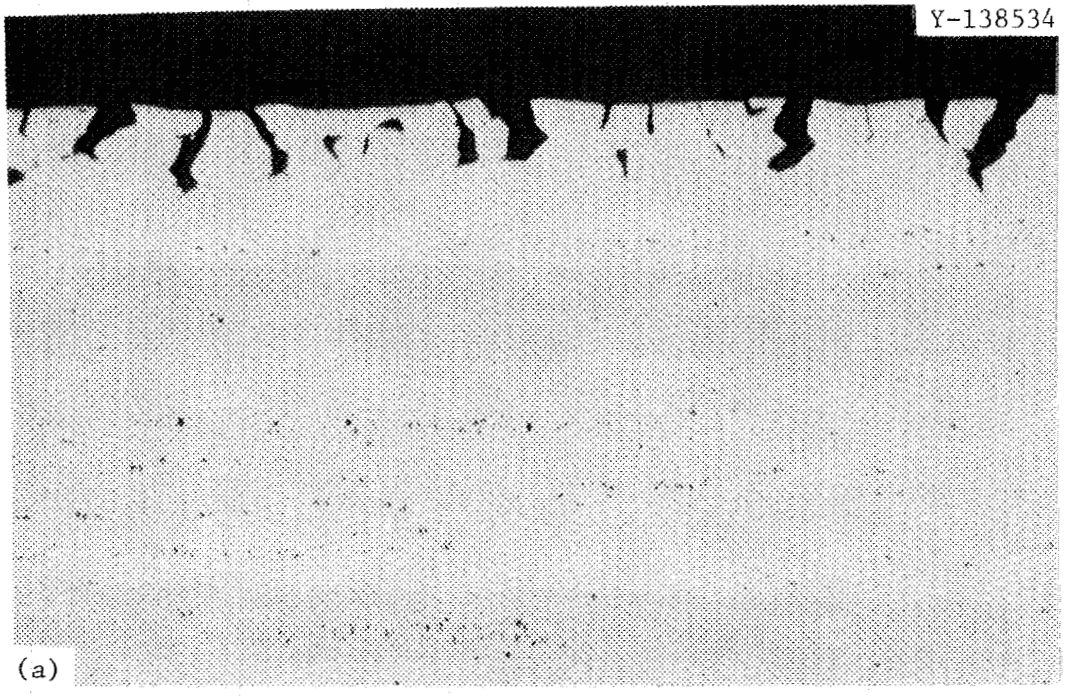
Fig. 9. Effect of Niobium in Modified Hastelloy N on Grain Boundary Cracking when Exposed in Salt–Cr_3Te_4 + Cr_5Te_6 for 250 hr at 700°C.

as a telluride rather than as elemental tellurium. Their calculations showed the critical potential for the formation of a telluride corresponded to a U(IV)/U(III) ratio of about 150 at 650°C.

To check this hypothesis, a salt pot was built, equipped with electrochemical probes, and filled with about 1.4 liters of MSBR fuel salt ($LiF-BeF_2-ThF_4-UF_4$). With the close cooperation of D. L. Manning, Analytical Chemistry Division, the oxidation potential and impurity content of the salt were monitored voltammetrically. Additions of $CrFe_2$ and about 1 g of Cr_3Te_4 were made, and a Hastelloy N specimen was exposed for 500 hr to salt with a U(IV)/U(III) ratio of about 90. Metallographic examination of the specimen after deformation showed extensive cracking (Fig. 10).

To lower the oxidation potential of the salt, a beryllium rod was immersed in the salt for an hour after which time the U(IV)/U(III) ratio had decreased to about seven. After another addition of CrF_2 and Cr_3Te_4 to the salt, another specimen was exposed to the mixture for 500 hr. A metallographic examination (Fig. 10) of the tensile tested specimen showed a complete absence of grain boundary cracks. These results were encouraging so a series of exposures was carred out over a wider range of U(IV)/U(III) ratios. Specimens were exposed for about 260 hr at U(IV)/U(III) ratios of approximately 10, 30, 60, 85, and 300. Photomicrographs of the gage section of the tensile specimens from this series of tests are shown in Fig. 11. As is evident in Fig. 12, a marked change occurred in the cracking behavior of Hastelloy N exposed in this system in going from a ratio of about 60 to about 100.

The results of the metallographic examination of the specimens exposed in the Cr-Te-U interaction experiment are collected in Table 4 and are given in the order of exposure. Before each exposure both $CrFe_2$ and Cr_3Te_4 (or $CrTe_{1.266}$) were added to the salt. According to the voltammetric measurements, the Cr^{++} content of the salt increased with successive $CrFe_2$ additions. As can be seen from Table 4, the first and eighth exposures were at about the same oxidation potential (90 and 85), but the cracking behavior of the two differed more than would be expected from the difference in exposure time and oxidation potential. This suggests that the difference in Cr^{++} concentration between the early and later exposures is an important factor that affects the cracking behavior, but no funding or plans exist



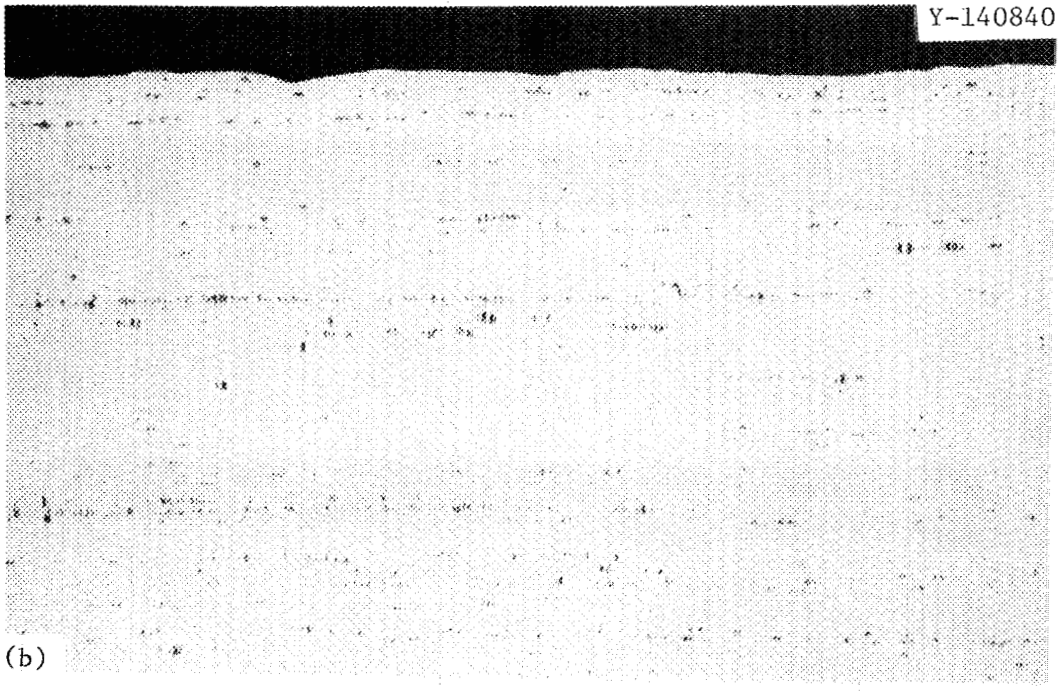


Fig. 10. Hastelloy N Exposed 500 hr at 700°C to MSBR Fuel Salt Containing $CrFe_2$ and Cr_3Te_4 With U(IV)/U(III) Equal to (a) 90 and (b) 7. 100×.

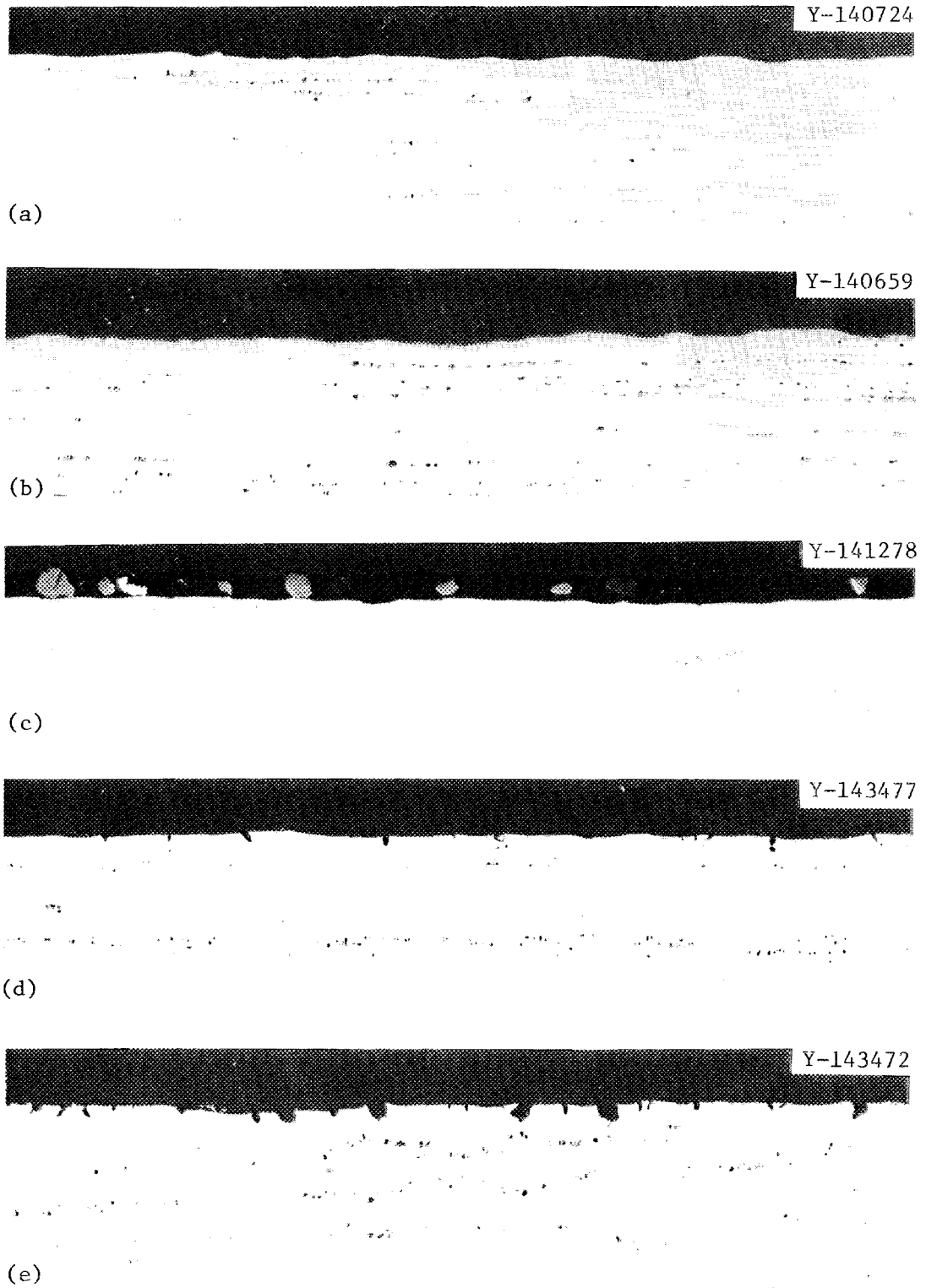


Fig. 11. Hastelloy N Exposed About 260 hr at 700°C to MSBR Fuel Salt Containing $CrFe_2$ and $CrTe_{1.266}$ with U(IV)/U(III) Equal to (a) 10, (b) 30, (c) 60, (d) 85, and (e) 300. 100×.

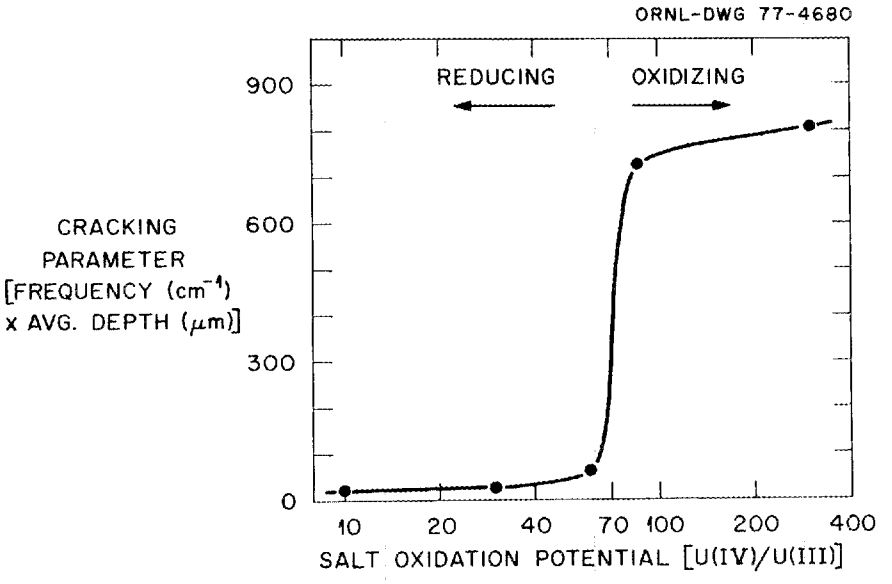


Fig. 12. Cracking Behavior of Hastelloy N Exposed 260 hr at 700°C to MSBR Fuel Salt Containing $CrTe_{1.266}$.

Table 4. Intergranular Crack Behavior of Hastelloy N Specimens Exposed in Cr-Te-U Interaction Experiment at 700°C

Specimen	Exposure Conditions			Crack Density (m^{-1})	Average Crack Depth (μm)	Density Times Depth
	Time (hr)	U(IV)/U(III)	Te Additive			
1	504	~90	Cr_3Te_4	12,200	74.8	0.9126
2	502	~7	Cr_3Te_4	0	0.0	0.0
3	257	~10	$CrTe_{1.266}$	200	3.6	0.0007
4	257	~10	$CrTe_{1.266}$	400	8.6	0.0034
5	257	~30	$CrTe_{1.266}$	200	12.7	0.0025
6	257	~30	$CrTe_{1.266}$	300	12.2	0.0037
7	257	~60	$CrTe_{1.266}$	500	13.0	0.0065
8	257	~85	$CrTe_{1.266}$	6,800	10.7	0.0728
9	262	~300	$CrTe_{1.266}$	6,000	13.6	0.0816

[a]All were standard Hastelloy N, heat 405065.

for further investigation of this possibility. (Also different was the telluride added, $CrTe_{1.333}$ vs $CrTe_{1.266}$, but from previous experience we would not expect it to make such a large difference).

Controlling the oxidation potential of the salt coupled with the presence of chromium ions in the salt appears to be an effective means of limiting tellurium embrittlement of Hastelloy N. However, further studies are needed to assess the effects of longer exposure times and to measure the interaction parameters for chromium and tellurium under varying salt oxidation potentials.

SUMMARY

As a result of these studies, we have found that Hastelloy N exposed in salt containing metal tellurides such as Li_xTe and Cr_yTe_z undergoes grain boundary embrittlement like that observed in the MSRE. The embrittlement is a function of the chemical activity of tellurium associated with the telluride. The degree of embrittlement can be reduced by alloying additions to the Hastelloy N. The addition of 1 to 2 at. % Nb significantly reduces embrittlement, but small additions of titanium or additions of up to 15 at. % Cr do not affect embrittlement.

We have found that if the U(IV)/U(III) ratio in fuel salt is kept below about 60, embrittlement is essentially prevented when $CrTe_{1.266}$ is used as the source of tellurium.

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