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MASS TRANSFER IN A 1370° C (2500° F) LITHIUM THERMAL-CONVECTION LOOP

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16. Abstract Experimental results from a test to evaluate interstitial element mass-transfer effects on T-111, ASTAR 811C, and ASTAR 1211C after 5000 hours in flowing lithium at 1370 ⁰ C (2500 ⁰ F) are presented. No gross corrosion effects were observed. However, hafnium and nitrogen transfer to cooler regions within the loop were noted. Oxygen was in general removed from test specimens, but there was no evidence to indicate that it was a major factor in the mass-transfer process. Carbon and hydrogen transfer were not detected.			
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SUMMARY

The results of materials compatibility tests on a thermal-convection loop constructed of the tantalum alloy T-111, containing lithium, and operated at a maximum temperature of 1370° C (2500° F) are described. The loop contained sheet test specimens of T-111 and the advanced tantalum alloys ASTAR 811C and ASTAR 1211C for the purpose of evaluating the effects of mass transfer of interstitial elements in flowing 1370° C (2500° F) lithium on these alloys. Test time was 5000 hours.

No gross corrosion effects were observed. Mass-transfer effects were most readily detected by specimen weight change measurements. Material had been removed from the hot region and deposited in the colder regions of the loop. A direct relation existed between post-test nitrogen content and weight change. Deposits with high hafnium content were found in the region of high nitrogen content by scanning electron microscopy. Oxygen was generally removed from the test specimens but did not have a major role in the mass-transfer process. Carbon and hydrogen transfer were not detected.

INTRODUCTION

The use of refractory-metal alloys in high-temperature space power systems has been of interest for many years. Tantalum alloys have been considered especially interesting because of their excellent fabricability and high strength at temperatures above 1000° C (°F). The presence of getter elements, such as hafnium, which chemically tie up oxygen has been shown to be beneficial in the control of refractory-metal alloy corrosion by alkali metals (refs. 1 and 2). The tantalum alloy T-111 (Ta-8W-2Hf) has received considerable attention as a typical high-strength alloy. A program to improve T-111 resulted in a series of alloys, of which ASTAR 811C (Ta-8W-1Re-1Hf-0.025C) and ASTAR 1211C (Ta-12W-1Re-1Hf-0.025C) are representative (refs. 3 and 4).

Although these alloys are generally considered compatible with alkali metals, interstitial element mass transfer with its associated corrosion effects has been a major cause

for concern in high-temperature operation.

This study was conducted primarily to evaluate mass transfer of interstitial elements in a few of the promising tantalum alloys when they are exposed to flowing 1370° C (2500° F) lithium. The alloys included in this study were T-111, ASTAR 811C, and ASTAR 1211C.

The loop was designed, fabricated, installed, and operated by the General Electric Company under NASA contract NAS 3-6474. The mass-transfer results were analyzed and evaluated at the NASA Lewis Research Center.

This report describes these materials compatibility test results. Although tensile specimens were included in the loop during test operation, this report covers only the materials compatibility aspects of the study. Mechanical property changes were not determined in this evaluation of the loop materials.

MATERIALS, APPARATUS, AND PROCEDURE

Loop Construction and Operation

This study was conducted to evaluate mass transfer of interstitial elements in T-111, ASTAR 811C, and ASTAR 1211C in flowing lithium. For this purpose a test loop of T-111 alloy tubing, 2.54 centimeters in outside diameter by 0.25 centimeter in wall thickness (1 in. by 0.1 in.), containing specimens of these alloys was designed, fabricated, and operated at 1370° C (2500° F) for 5000 hours (refs. 5 to 7).

Test Materials

Two types of specimens were tested. The sheet tensile specimen design shown in figure 1 permitted removal of chemical and metallographic samples after the specimens were exposed to flowing lithium under the test conditions. Removal of these samples left a standard tensile specimen for determination of possible strength changes caused by mass transfer of interstitial elements or possible structural changes.

Sheet specimens of T-111 and ASTAR 1211C were also machined to the dimensions shown in figure 2. These specimens were used for chemical and metallographic evaluation of these alloys.

Interstitial chemistry of the materials used to fabricate these test specimens is presented in table I. Metallographic structures of these materials are shown in figure 3. All test specimens were to have been recrystallized, followed by a 1-hour, 1316° C (2400° F) anneal. Inadvertently, the T-111 tensile specimens were tested in the cold-

worked condition.

The distribution of T-111, ASTAR 811C, and ASTAR 1211C specimens in the loop is shown schematically in figure 4, along with the average test conditions.

ANALYTICAL RESULTS

Visual

When they were removed from the loop, all specimens appeared in good condition with no visual indications of gross corrosion or deterioration. All the T-111 specimens appeared bright, shiny, and unchanged by the test exposure. Some of the ASTAR 811C and ASTAR 1211C specimens were dull gray in color and were not as bright and shiny as the pretest material.

Chemistry

All specimens were weighed to determine weight change caused by the test. The results are given in table II and in figure 5. The T-111 specimens lost weight slightly at all locations in the loop, but lost more in the hotter regions. The ASTAR alloys lost weight in the hotter regions of the loop and gained weight in the cooler regions.

Selected specimens were evaluated using chemical, metallographic, and scanning electron microscopic (SEM) examination. The chemical analyses are given in table II. These values were used in plotting the curves in figure 5. The values for hydrogen and carbon seemed to be randomly scattered, with no apparent trends with position in the loop. Therefore, these data were not plotted.

Both ASTAR 1211C and T-111 exhibited oxygen loss at all locations. The analyses of ASTAR 811C specimens indicated a possible slight oxygen gain in the cooler regions and an oxygen loss in the hotter regions.

In all cases the nitrogen concentrations were higher in specimens from the cooler regions of the loop than in those from the hotter regions. When viewed with respect to the significant weight changes observed, the nitrogen mass transfer far outweighs any possible effect oxygen mass transfer might have.

Metallography

The metallographic results for T-111, ASTAR 811C, and ASTAR 1211C specimens are shown in figures 6 to 9. These materials went through varying amounts of structural change during exposure, depending on their location in the loop.

The T-111 tensile and chemical-metallographic specimens exhibited recrystallization, grain growth, and a general cleaning of the grains. These effects were observed throughout the specimen thickness. Their extent varied with the temperature of lithium exposure, being greater at the higher temperatures.

The ASTAR 811C specimens tested at the lower temperatures seemed to have more precipitates than the pretest material. The metallography on specimens tested at the higher temperatures showed generally cleaner grains, grain growth at the surface exposed to lithium, and a tendency for the precipitates to coalesce at the grain boundaries.

Metallography did not reveal any real differences between the pretest and lower temperature post-test specimens. Specimens exposed at the higher temperatures, however, showed a general precipitate coalescence at grain boundaries and a layer of cleaner grains at the surface exposed to lithium.

The only corrosive effects of lithium exposure seemed to be a slight attack on the surface of T-111 specimens in the hot regions of the loop. ASTAR 811C and ASTAR 1211C were not corrosively attacked under these conditions.

Scanning Electron Microscopy

SEM photomicrographs and spectral scans were taken of surfaces of selected test specimens. The T-111 specimens are shown in figure 10. The as-prepared specimens had the typical abraded and chemically cleaned surface. The as-tested surfaces, however, were cleaned by the lithium as though by a chemical etch, with grain structure clearly delineated. Only on one specimen did it appear that a minute amount of deposition might have occurred. Spectral scans in all cases yielded a typical spectrum for the T-111 composition. Only one of the specimens examined showed a possibility of a second phase at a few of the grain boundaries. Spectral scans indicated this phase to be of higher hafnium and lower tantalum content than the bulk grains.

Results of SEM on selected ASTAR 811C specimens are shown in figure 11. The as-prepared specimen showed the results of abrading and chemical cleaning. Specimen A-T-4, taken from the end of the hot leg, and specimen A-T-10, taken from the end of the cold leg, clearly showed the typical etched appearance of material exposed to hot lithium. However, many particles were observed on the grain surfaces and at grain boundaries. Spectral scans of these particles did not reveal any observable compositional differences from the bulk grains. Specimen A-T-6, taken from the beginning of the cold leg, how-

ever, was in marked contrast to the others. There was an obvious dense, angular, crystalline material covering the surface of the sample. A spectral scan of this surface indicated hafnium to be the main metallic constituent of this material.

The results of SEM on selected ASTAR 1211C samples are shown in figure 12. The as-prepared specimen exhibited a highly overetched, pitted surface. Specimen A-C-2, taken from just beyond the heater exit, exhibited a highly etched, but unpitted, surface similar to that of ASTAR 811C. The surface was also covered with very fine particles. Spectral scans failed to show any compositional difference between ASTAR 1211C and the particles. The surface of specimen A-C-11, taken from just beyond the cold-leg exit, was covered with a dense, angular, crystalline deposit very similar to that of ASTAR 811C specimen A-T-6. These deposits masked the surface grain structures of both specimens. A spectral scan of this surface also indicated the main metallic constituent to be hafnium.

DISCUSSION OF RESULTS

The similarities between the weight change and nitrogen content curves in figure 5 are striking. The weight changes of all specimens correspond to the observed nitrogen concentration changes. Therefore, the major interstitial mass-transfer effect is concluded to be nitrogen transfer. It is further obvious that the observed nitrogen mass transfer could not have resulted from only the nitrogen initially in the test specimens. The T-111 which comprised the loop wall structure also contained nitrogen and was probably the major nitrogen source.

The lithium exposure resulted in oxygen loss for all test specimens except the ASTAR 811C specimens in the cooler regions of the loop. These had a maximum increase in oxygen content of only 10 ppm. It would appear that oxygen mass transfer from one alloy to another around the loop was slight. The main transfer of oxygen seems to have been from the alloys into the lithium stream. Because the changes in oxygen content were not consistent with the observed weight changes and the oxygen contents were maintained at low levels, oxygen transfer was concluded not to be a major factor in the mass-transfer process.

The fact that lithium generally removed oxygen from these materials is important in another respect. It means that the oxygen content of the liquid lithium in an actual system can increase greatly, which can increase the likelihood of plugging small passages with impurities. Thus, it is important to have impurity monitoring and removal devices on any actual system that uses circulating lithium.

Mass transfer of carbon and hydrogen apparently did not occur. Analyses for these elements showed no trend with loop position. The variations observed were probably normal scatter inherent in alloy inhomogeneities and in the analytical techniques for these

elements.

Basically, two different types of deposits were observed on the test specimens. Dense, angular, crystalline deposits occurred in those regions of the loop which experienced a net heat loss. These were probably deposited during the test. The second type of deposit was the fine particles found on several specimens. These fine particles were found on specimens located both in regions of the loop which were being heated and in regions which were losing heat. It appears likely, therefore, that these fine particles were a result of a deposition process occurring during test shutdown. Since neither type of deposit was found on the T-111 specimens, it must be concluded that the ASTAR alloys have sites which are more favorable for deposition of these materials from lithium solution than does T-111.

The chemical analyses and the SEM spectral scans of the dense crystalline deposits show them to be, probably, either hafnium nitride particles or hafnium crystals with a high nitrogen content.

The mass-transfer effects observed as a result of this test were minor in that no serious, gross corrosion results were observed. Only some mass transfer of hafnium and nitrogen was identified. Therefore, from a corrosion and mass-transfer standpoint these gettered tantalum alloys are acceptable construction materials for high-temperature lithium systems. However, the question of retention of short-term tensile and long-term creep properties is yet unanswered. These strength properties of tantalum alloys after exposure to lithium must be investigated before a final decision on system acceptability can be made.

CONCLUSIONS

The conclusions drawn from this test, which exposed T-111, ASTAR 811C, and ASTAR 1211C specimens to flowing lithium in a thermal-convection loop constructed of T-111 and operated at a maximum temperature of 1370^o C (2500^o F) for 5000 hours, are as follows:

1. From a corrosion standpoint, the gettered tantalum alloys are acceptable construction materials for high-temperature lithium systems. Corrosion or mass-transfer effects were minor under the test conditions used in this study.
2. Minor mass transfer of hafnium and nitrogen resulted in deposits of hafnium nitride or hafnium with a high nitrogen content on ASTAR 811C and ASTAR 1211C in regions of high net heat loss.

3. Oxygen transfer was not a major factor in the mass-transfer process under these test conditions.

4. Carbon and hydrogen mass transfer did not occur at these test conditions.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 1, 1973,
501-21.

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TABLE I. - INTERSTITIAL CHEMISTRY OF
STARTING TEST MATERIAL

Alloy	Oxygen	Nitrogen	Carbon	Hydrogen
	Content, ppm			
T-111 tensile specimens	30	9	8	---
T-111 chemical-metallographic specimens	50	4	18	0.4
ASTAR 811C specimens	17	4	194	---
ASTAR 1211C specimens	136	8	270	.7

TABLE II. - WEIGHT CHANGE AND CHEMICAL

ANALYSES OF TESTED SPECIMENS

Specimen	Distance from heater inlet, cm	Temperature, °C	Weight change, mg/cm ²	Chemical content, ppm			
				Oxygen	Nitrogen	Carbon	Hydrogen
T-111							
T-T-2	6.0	----	-0.28	--	---	---	---
T-T-3	20.0	----	-.52	4	2	9	---
T-T-4	34.0	----	-.18	4	<2	8	---
T-T-5	48.0	----	-.62	2	<2	20	---
T-C-1	60.0	----	-.87	13	3	61	1.0
T-C-2	62.5	----	-.80	--	---	---	---
T-C-3	65.0	----	-.93	--	---	---	---
T-C-4	77.0	----	-.89	14	1	61	1.2
T-C-5	79.5	----	-.69	--	---	---	---
T-C-6	82.0	----	-.82	--	---	---	---
T-C-7	93.5	----	-.77	12	3	62	1.2
T-C-8	96.0	1330	-.80	--	---	---	---
T-C-9	98.5	----	-.86	--	---	---	---
T-T-6	109.5	----	-.75	2	2	18	---
T-T-7	124.0	1285	-.55	4	<2	10	---
T-T-8	138.0	----	-.51	--	---	---	---
T-T-9	152.0	1220	-.34	5	4	11	---
T-T-10	166.0	1205	-.26	--	---	---	---
T-C-10	178.0	----	-.30	12	16	55	.8
T-C-11	180.5	----	-.30	--	---	---	---
T-C-12	183.0	----	-.30	--	---	---	---
T-C-13	194.0	----	-.21	10	14	50	1.2
T-C-14	197.0	----	-.34	--	---	---	---
T-C-15	200.0	----	-.23	--	---	---	---
T-C-16	210.5	----	-.17	16	16	26	.6
T-C-17	213.5	----	-.25	--	---	---	---
T-C-18	216.0	1165	-.32	9	10	18	.2
T-T-1	229.0	----	-.13	8	9	17	---
ASTAR 811C							
A-T-2	12.5	----	0.44	16	30	195	---
A-T-3	27.0	----	-.13	6	2	183	---
A-T-4	41.5	----	-.18	5	<2	147	---
A-T-5	55.5	1360	-.27	5	<2	172	---
A-T-6	116.5	----	-.14	<2	<2	195	---
A-T-7	130.5	----	-.07	3	<2	148	---
A-T-8	145.0	----	.39	14	20	201	---
A-T-9	158.5	----	2.35	26	74	228	---
A-T-10	173.0	----	3.72	18	112	190	---
A-T-1	236.0	1220	3.51	25	163	220	---
ASTAR 1211C							
A-C-1	68.0	1350	-0.42	11	3	348	2.1
A-C-2	71.0	----	-.45	--	---	---	---
A-C-3	74.0	----	-.32	--	---	---	---
A-C-4	84.5	----	-.30	6	2	326	.8
A-C-5	87.0	----	-.41	--	---	---	---
A-C-6	90.5	----	-.31	--	---	---	---
A-C-7	101.5	----	-.35	10	1	353	1.4
A-C-8	105.0	----	-.36	--	---	---	---
A-C-10	185.5	1185	5.36	26	106	367	1.6
A-C-11	188.0	1185	5.51	--	---	---	---
A-C-12	191.0	----	5.89	--	---	---	---
A-C-13	202.0	----	5.13	26	105	320	1.6
A-C-14	205.0	----	5.04	--	---	---	---
A-C-15	208.0	----	5.35	--	---	---	---
A-C-16	218.5	----	5.30	23	117	398	2.0
A-C-17	221.5	----	5.62	--	---	---	---
A-C-18	224.0	----	5.49	18	142	245	.4

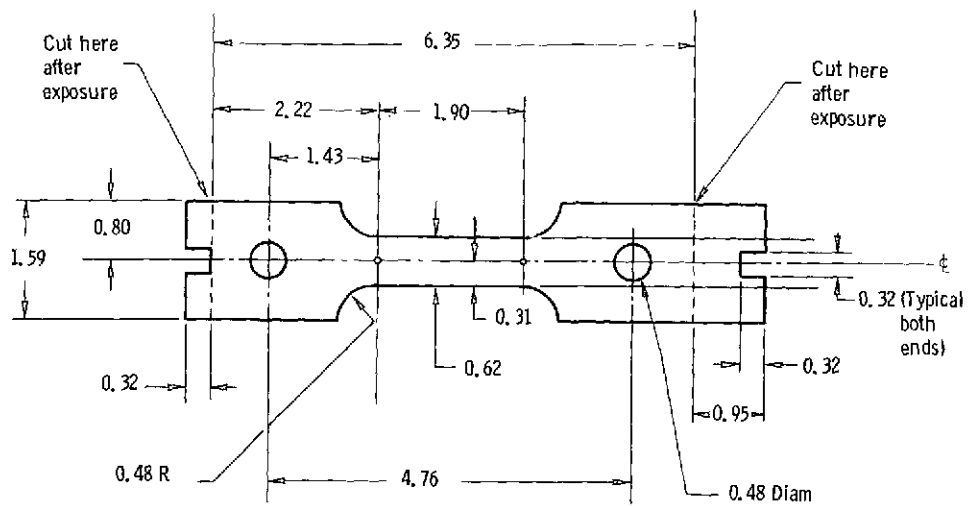


Figure 1. - Design of special sheet tensile specimen for insertion in vertical test sections of lithium thermal-convection test loop. (All dimensions are in cm.)

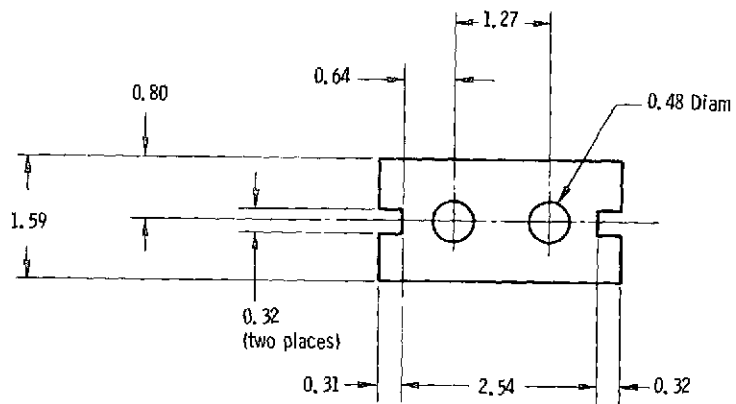
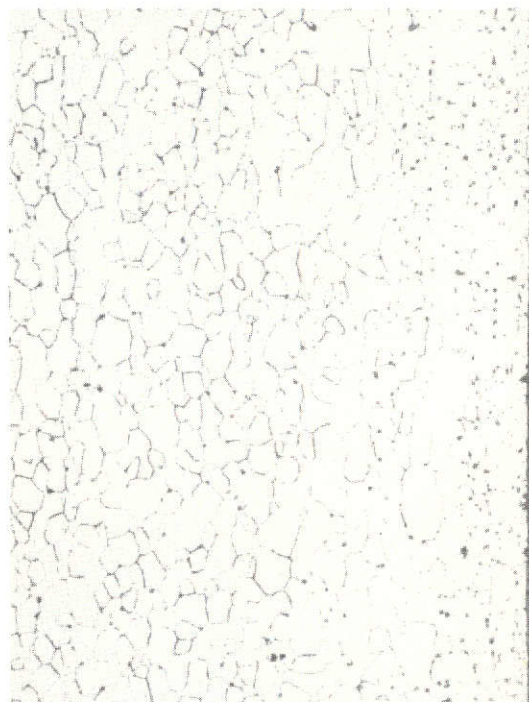


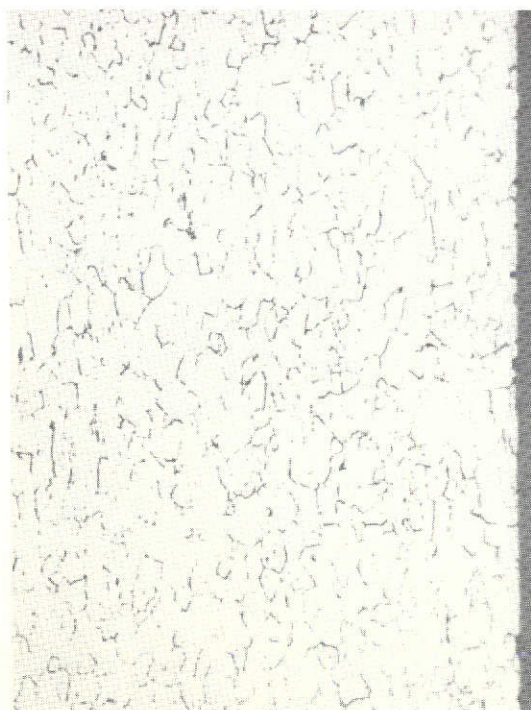
Figure 2. - Design of sheet test specimen for insertion in curved test sections of lithium thermal-convection test loop. (All dimensions are in cm.)



(a) T-111 tensile specimen sheet.



(b) T-111 chemical and metallographic specimen sheet.



(c) ASTAR 811C specimen sheet.



(d) ASTAR 1211C specimen sheet.

Figure 3. - Structure of materials used for test specimens.

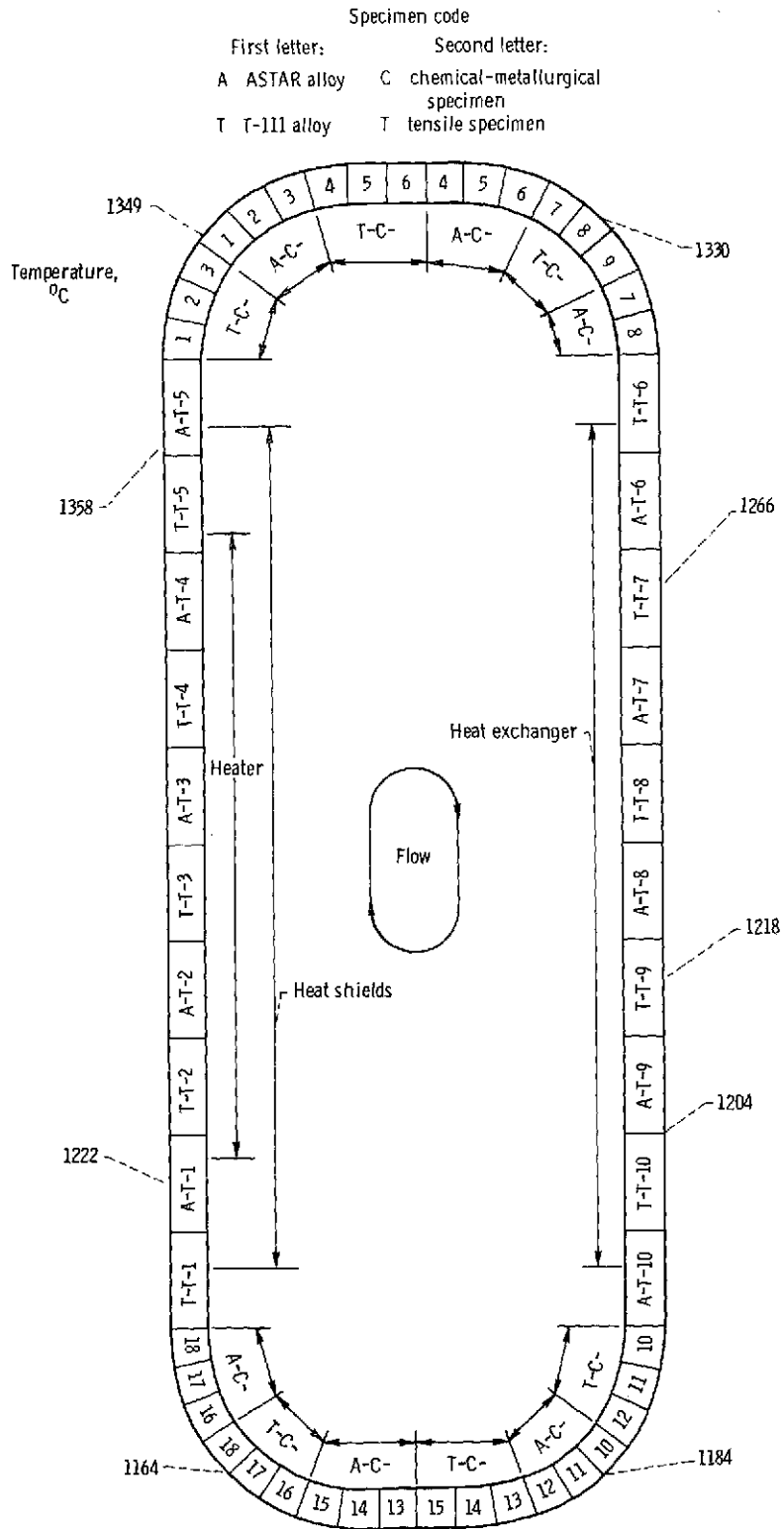


Figure 4. - Schematic diagram of the test loop showing specimen locations and average test temperatures. Total time, 5038 hours; chamber pressure, 6×10^{-9} torr.

Specimen	Nitrogen	Oxygen
	Approximate initial concentration, ppm	
T-111 tensile specimens	10	50
T-111 chemical-metallographic specimens	10	30
ASTAR 811C	5	15
ASTAR 1211C	5	135

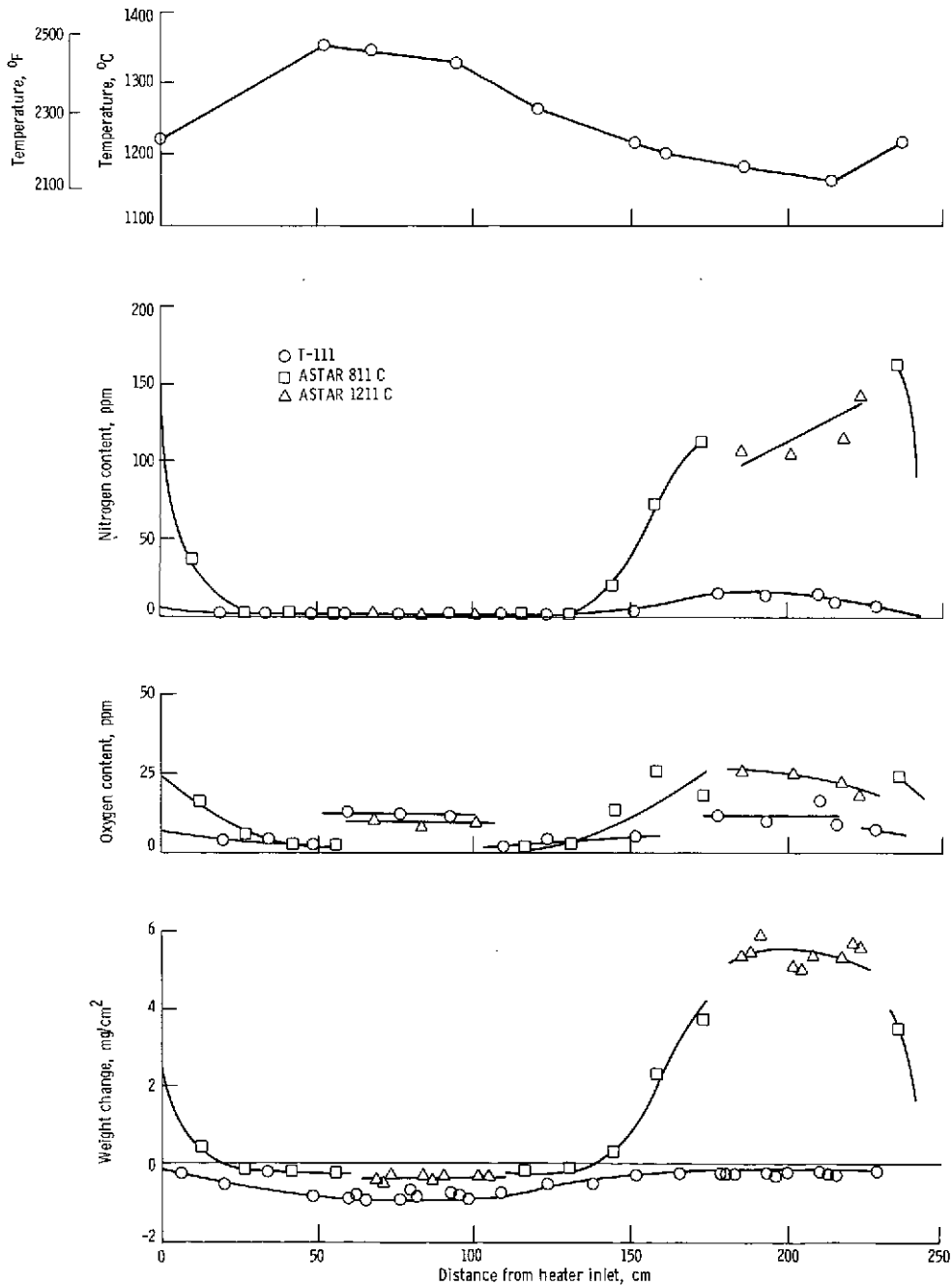
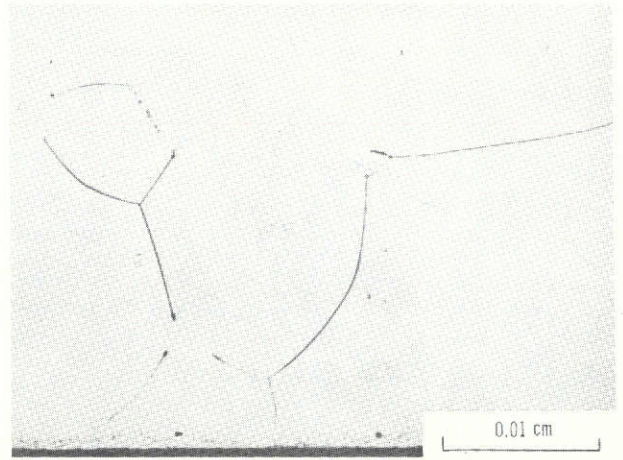


Figure 5. - Weight change, specimen oxygen and nitrogen content, and temperature as functions of distance from heater inlet.



(a) Specimen T-T-1, taken from heater inlet, about 1175° C (2150° F).

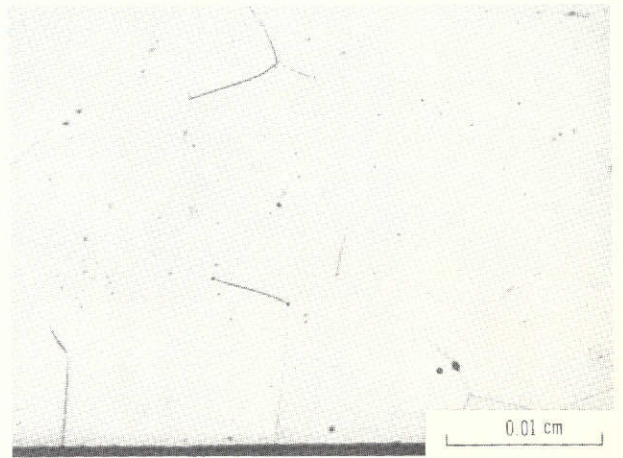


(b) Specimen T-T-5, taken from heater outlet, about 1365° C (2490° F).

Figure 6. - Photomicrographs of T-111 tensile specimens after testing in loop.

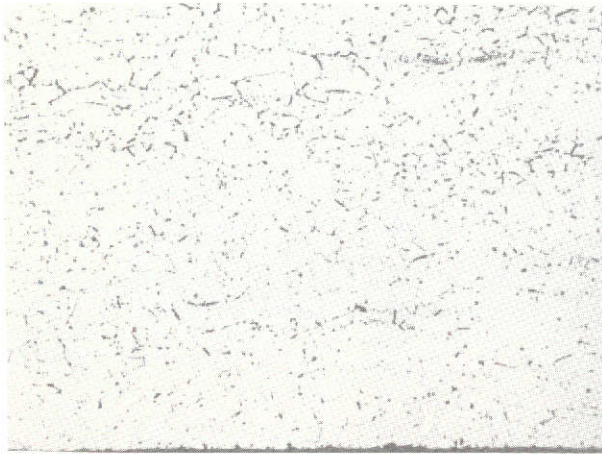


(a) Specimen T-C-16, taken from near heater inlet, about 1165° C (2130° F).



(b) Specimen T-C-1, taken from near heater outlet, about 1350° C (2460° F).

Figure 7. - Photomicrographs of T-111 chemical-metallographic specimens after testing in loop.

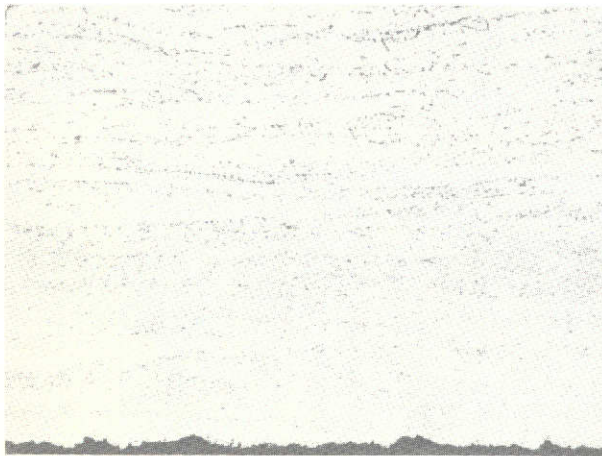


(a) Specimen A-T-9, taken from near cooling heat-sink exit, about 1215° C (2220° F).

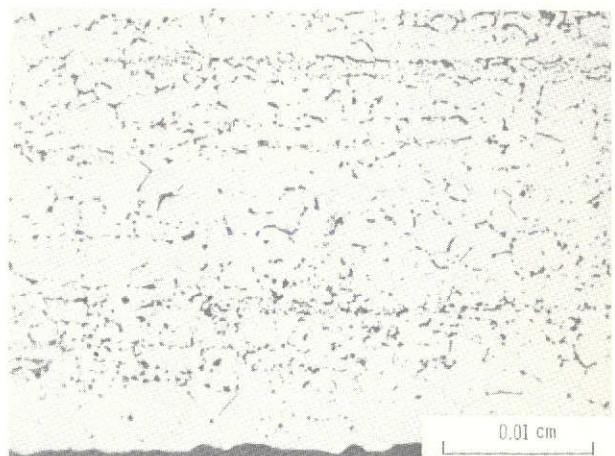


(b) Specimen A-T-5, taken from heater exit, about 1360° C (2475° F).

Figure 8. - Photomicrographs of ASTAR 811C specimens after testing in loop.

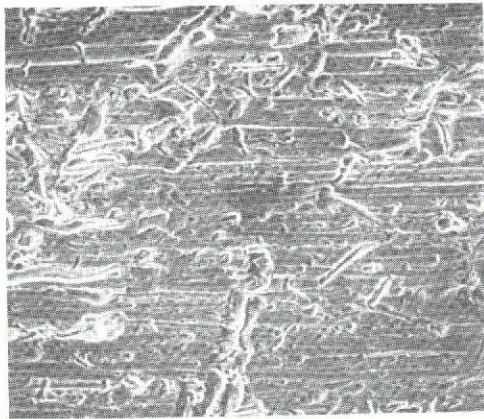


(a) Specimen A-C-16, taken from near heater inlet, about 1165° C (2130° F).

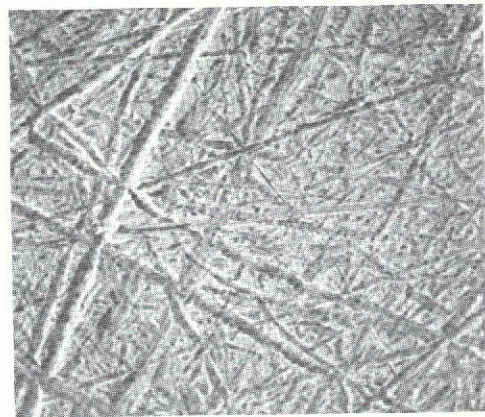


(b) Specimen A-C-1, taken from near heater exit, about 1350° C (2460° F).

Figure 9. - Photomicrographs of ASTAR 1211C specimens after testing in loop.



(a-1) As-cleansed, prior to test.



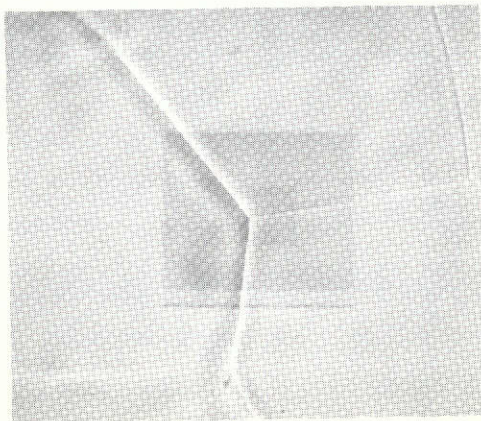
(b-1) As-cleansed, prior to test.



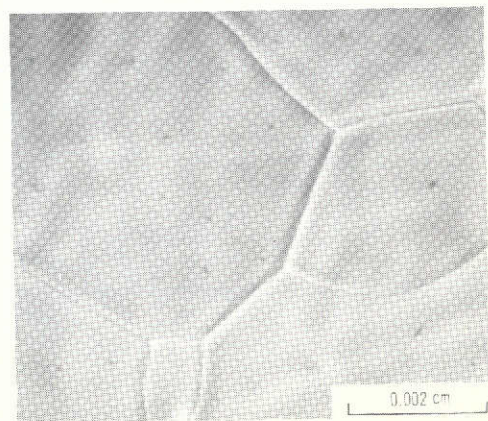
(a-2) Specimen T-T-10, exposed to lithium at about 1205° C (2200° F).



(b-2) Specimen T-C-11, exposed to lithium at about 1190° C (2170° F).



(a-3) Specimen T-T-6, exposed to lithium at about 1320° C (2410° F).

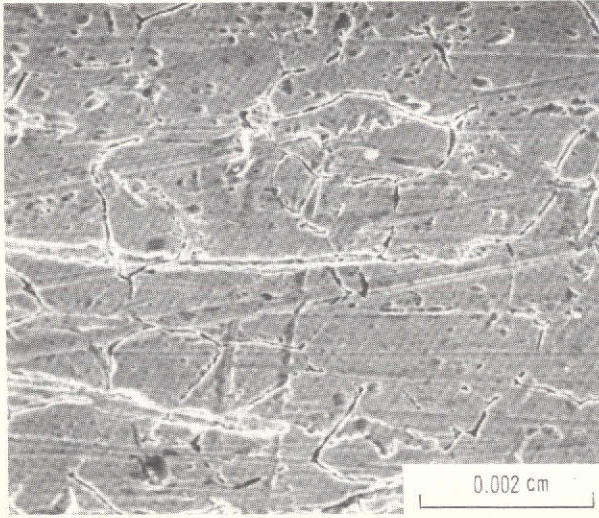


(b-3) Specimen T-C-2, exposed to lithium at about 1350° C (2460° F).

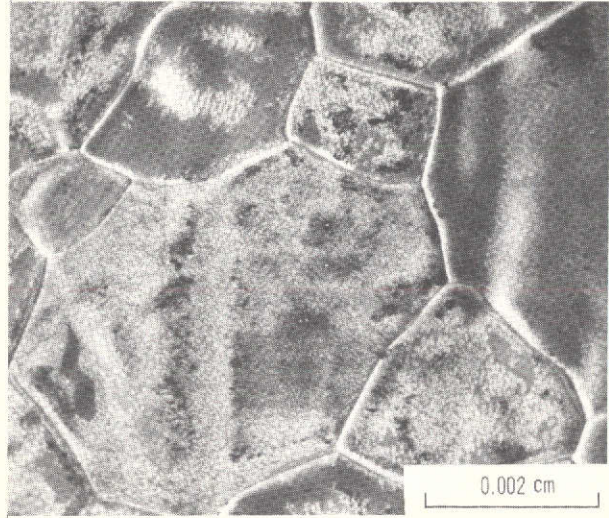
(a) Tensile specimen surfaces.

(b) Chemical-metallographic specimen surfaces.

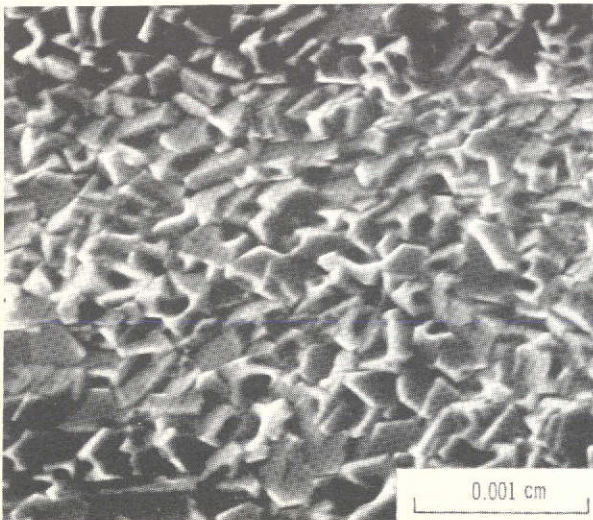
Figure 10. - SEM photomicrographs of T-111 tensile and chemical-metallographic specimen surfaces. Voltage, 25 kilovolts; tilt, 20°.



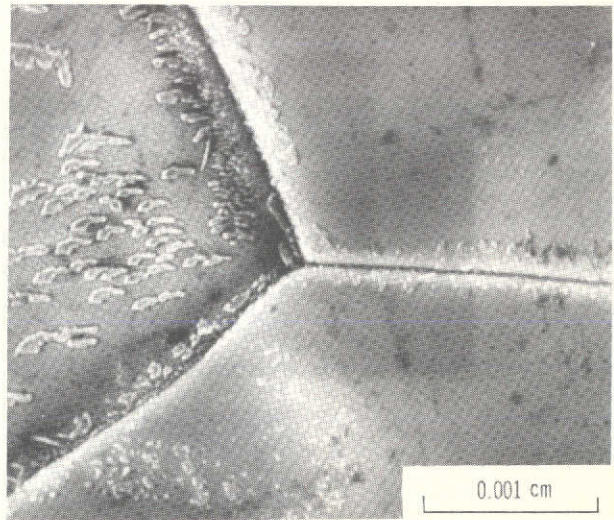
(a) As-cleaned, prior to test.



(b) Specimen A-T-10, exposed to lithium at about 1190° C (2170° F).

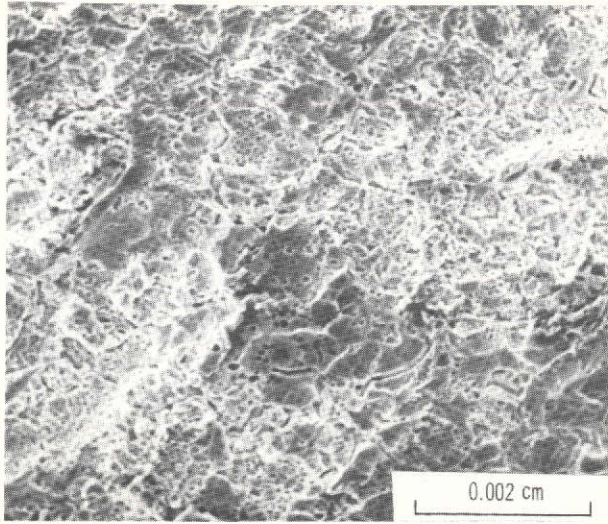


(c) Specimen A-T-6, exposed to lithium at about 1310° C (2390° F).

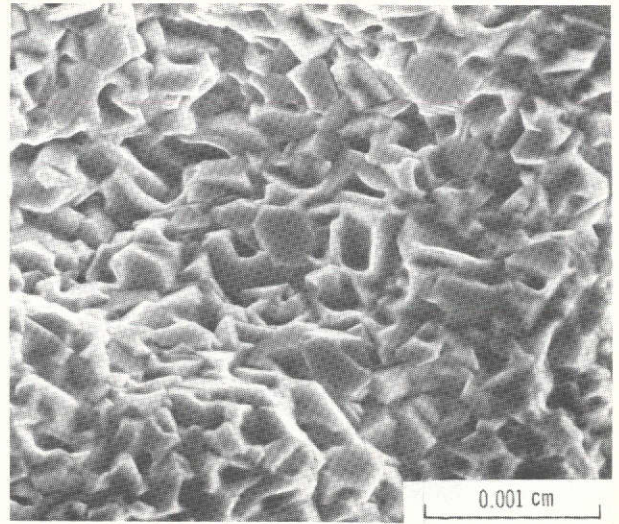


(d) Specimen A-T-4, exposed to lithium at about 1325° C (2420° F).

Figure 11. - SEM photomicrographs of ASTAR 811C specimen surfaces. Voltage, 25 kilovolts; tilt, 20°.



(a) As-cleaned, prior to test.



(b) Specimen A-C-11, exposed to lithium at about 1185° C (2170° F).



(c) Specimen A-C-2, exposed to lithium at about 1350° C (2460° F).

Figure 12. - SEM photomicrographs of ASTAR 1211C specimen surfaces. Voltage, 25 kilovolts; tilt, 20°.