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TECHNIQUES FOR LITHIUM REMOVAL FROM 1040° C AGED TANTALUM ALLOY, T-111

by Randall F. Gahn

Lewis Research Center
Cleveland, Ohio 44135

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16. Abstract

The liquid ammonia and vacuum distillation techniques were found to be satisfactory for removing lithium from 1040° C aged T-111 (tantalum - 8-percent tungsten - 2-percent hafnium). Results of ductility tests and chemical analysis show that these two methods are adequate for removing lithium without embrittlement or contamination of the T-111. Moist air exposure of T-111 with traces of lithium on the surface produced mixed results. Some specimens were ductile; others were brittle. Brittle T-111 had an increased hydrogen content. Water removal of lithium from T-111 caused brittleness and an increased hydrogen concentration.

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TECHNIQUES FOR LITHIUM REMOVAL FROM 1040° C AGED TANTALUM ALLOY, T-111

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SUMMARY

Studies were made to determine the effectiveness of the liquid ammonia and vacuum distillation techniques for removing lithium from T-111 (a tantalum alloy containing, nominally, 8 percent tungsten and 2 percent hafnium), which had been heated for 100 hours at 1040° C. Lithium was removed from tube and sheet specimens by dissolution in freshly condensed ammonia or by vaporizing it at 700° C in vacuum. The T-111 specimens were evaluated by bend testing and by chemical analyses. All specimens that had lithium removed by either technique were ductile and showed no significant changes in oxygen, nitrogen, or hydrogen concentration.

The effects of water removal of lithium and moist air exposure were investigated to determine the susceptibility of T-111 to contamination. Specimens that were cleaned with water were brittle and showed an increase in hydrogen content. When T-111 with surficial traces of lithium was exposed to moist air, some specimens were ductile, some were brittle. Brittle samples had an increased hydrogen concentration. The results of this study show that care is necessary in lithium removal to avoid hydrogen embrittlement of the T-111.

INTRODUCTION

In advanced space nuclear power systems it is expected that alkali metals will be employed as heat-transfer fluids, while refractory metals such as tantalum will be used for their containment. The tantalum alloy, T-111 (tantalum - 8 percent tungsten - 2 percent hafnium) has been studied extensively for space-power applications. Brittle, intergranular cracking has been observed in room-temperature ductility tests on T-111 specimens aged at 1040° C for long periods of time. This problem was first encountered during the evaluation of a 1040° C portion of a 10 000-hour potassium/T-111 corrosion loop test (ref. 1), and later it was encountered on T-111 fuel pins exposed to lithium at 1040° C (ref. 2).

In a study on long time, 1040° C aged T-111 to determine the cause of this embrittlement (ref. 3), it was found that aging caused T-111 to become highly sensitive to hydrogen pickup, apparently because of hafnium segregation at grain boundaries. It was concluded that embrittlement was due to hydrogen pickup from exposure of the aged T-111 to a source of moisture during post-age handling and testing.

Hydrogen can also be introduced into refractory metals during cleaning after alkali metal exposure (refs. 4 and 5). Alkali metal removal solvents such as water and alcohol liberate hydrogen by their reaction with alkali metals and should be avoided. Two methods that do not liberate hydrogen during alkali metal removal are vacuum distillation and reaction with liquid ammonia. Previous studies (refs. 4 and 5) on unaged T-111 or T-111 aged at temperatures other than 1040° C have shown that there is no hydrogen contamination of T-111 when using either of these methods, although improper use of either technique can lead to hydrogen contamination. Inherent in these two methods is the possibility of nitrogen or oxygen contamination. Vacuum distillation removal requires heating of the T-111 at 700° C for several hours in vacuum. Liquid ammonia removal requires the specimens to be subjected to a highly reactive environment.

The purpose of this study was to determine if the liquid ammonia and vacuum distillation techniques are adequate for removing lithium from 1040° C aged T-111 without hydrogen, oxygen, and nitrogen contamination. Tubing and sheet specimens were exposed to lithium for 100 hours at 1040° C, and the lithium removed by the liquid ammonia or vacuum distillation techniques. Ductility tests and chemical analyses were conducted on the specimens to determine if contamination had occurred during lithium removal. To serve as baseline data, similar tests were conducted on as-received T-111, on T-111 heat treated for 2 hours at 1316° C, and on T-111 aged for 100 hours at 1040° C without lithium. Studies were also made to determine the susceptibility of the T-111 to interstitial element contamination during water removal of the lithium and during exposure to moist air.

EXPERIMENTAL PROCEDURE

Test Capsule Preparation

Five T-111 capsules, 6.35-centimeter long (2.5-in.) by 1.27-centimeter (0.500-in.) outside diameter by 0.089-centimeter (0.035-in.) wall thickness, were made by electron beam welding 0.076-centimeter (0.030-in.) thick disks to one end of each of five T-111 tubes. T-111 sheet specimens 2.54-centimeter (1.00-in.) long by 0.64-centimeter (0.25-in.) wide by 0.051-centimeter (0.020-in.) thick were sheared, and holes were drilled at the ends of each specimen. The capsules and sheet specimens were then cleaned using the following procedure:

(1) Liquid and vapor degrease in freon precision cleaning agent

(2) Ultrasonic clean in detergent

(3) Acid etch in a mixture of nitric acid, sulfuric acid, hydrofluoric acid, and water (1:2:1.5:4.5 parts by volume, respectively)

(4) Ultrasonic rinse in distilled water and dry

The sheet specimens were tied with tantalum wire into bundles containing seven specimens. Tantalum spacers (0.025-cm (0.010-in.) thick) separated the specimens. The sheet specimens and capsules were annealed at 1316° C for 2 hours at 1.3×10^{-5} newton per square meter (1×10^{-7} torr) before assembly. A capsule with the specimens is shown in figure 1.

Capsules and specimens were placed into an electron-beam welding chamber, and the system was evacuated to 2.7×10^{-4} newton per square meter (2×10^{-6} torr). The capsules and lithium were heated to 260° C and 1.2 grams of molten lithium was dispensed into four of the five capsules. While the lithium was still liquid, a bundle of sheet specimens was put into each capsule. After lithium solidification, the lids were put in place, and the capsules were electron beam welded closed. The capsules were given an acid etch before installation in the vacuum furnace to remove handling contamination. Three capsules, two with lithium and one without, were heated in an ion pumped vacuum system at about 5.3×10^{-6} newton per square meter (4×10^{-8} torr) for 100 hours at 1040° C (1900° F). The other two lithium-filled capsules were given no further heat treatment. T-111 specimens were attached outside the capsules to evaluate the quality of the vacuum. Interstitial analysis of these specimens following the test showed no significant contamination, indicating that the vacuum level was satisfactory.

Lithium Removal

The capsules were opened in an argon atmosphere glove box using a hacksaw. The lithium was melted; the bundles were removed; and the bulk lithium was siphoned from the capsule. Bundles were left intact for distillation removal, but separated for the liquid ammonia, moist air, and water treatments. Ring specimens were cut from each capsule using a hacksaw. The ring and sheet specimens were then placed in capped vials or the distillation transfer chamber and taken to the appropriate lithium removal apparatus.

Liquid ammonia treatment. - Liquid ammonia was prepared by condensing gaseous ammonia at -60° C and collecting the liquid in dewar flasks. The specimens were removed from the capped vials, immersed in the liquid ammonia, and agitated for about 5 minutes. The solution developed the characteristic deep blue color from the dissolution of the lithium in the liquid ammonia. The specimens were then given two rinses in fresh liquid ammonia. Both rinse solutions remained colorless, which indicated complete lithium removal. Immediately following the last rinse, the specimens were recapped in glass vials.

Vacuum distillation treatment. - The specimens, sealed in the transfer chamber, were taken to the distillation apparatus. In vacuum the specimens were remotely transferred to the distillation holder. Lithium was removed from the specimens by heating the holder to 700° C for 1 hour at a system pressure of about 1.3×10^{-3} newton per square meter (1×10^{-5} torr). After distillation the specimens were placed in the transfer chamber and returned to the dry box.

Moist air exposure. - Lithium was oxidized from the surface of several specimens by exposing them to a 100 percent relative humidity atmosphere. Following exposure to the moist air for at least 1 week, the specimens were removed, rinsed with distilled water to remove the lithium hydroxide, dried, and prepared for evaluation.

Water removal. - Lithium was removed from several specimens by submerging the specimen in water. The specimens were then rinsed, dried, and evaluated.

Specimen Preparation and Evaluation

Following the lithium removal treatment, the samples were prepared for ductility testing. Since airborne moisture previously had been shown to cause brittleness in postage testing of long time, 1040° C aged T-111 (ref. 3), ductility tests for this study were done both in a dry argon atmosphere and in ambient air to determine if the same moisture reaction occurred on T-111 aged at 1040° C for 100 hours. Ring specimens were prepared by filing and dry sanding them with 280- and 400-grit silicon carbide paper. The ring samples were tested by flattening in a vise until the opposite walls made contact. Bend tests were conducted on sheet specimens by completely bending the sheet back on itself around a 0.025-centimeter (0.010-in.) radius. Bends were made on both ends of the sheet specimen. Typical tube and sheet specimens following ductility testing are shown in figure 2.

Following the ductility tests, the specimens were sectioned and analyzed chemically and metallographically. Chemical analysis for oxygen, nitrogen, and hydrogen was done by vacuum fusion. Metallographic specimens were etched and examined for second phases and cracks.

RESULTS AND DISCUSSION

The results of ductility tests and chemical analyses on the T-111 specimens following the various heat treatments and lithium removal conditions are summarized in tables I and II. Each sheet specimen provided two bend tests (one at each end of the specimen) and each bend test is recorded separately. The discussion of the results is presented according to the lithium removal conditions and in the same order as listed in tables I and II.

Lithium Absent - No Removal Treatment

Tests were made on specimens in the as-received condition, after vacuum annealing for 2 hours at 1316° C, and after vacuum aging for 100 hours at 1040° C to determine if the heat treatment in the absence of lithium affected the ductility or interstitial element concentrations. All sheet specimens, regardless of condition, were ductile both in ambient air and in dry argon. The as-received tube specimens and the tube specimens annealed in vacuum at 1316° C also were ductile. Two of the tubing specimens, aged at 1040° C for 100 hours, developed edge cracks during flattening in dry argon. The third tube specimen prepared and tested in air was ductile. These results indicate that no significant embrittlement occurred as a result of heat treatment without lithium.

Ductility testing of the 100-hour aged T-111 specimens in ambient air did not cause embrittlement as it did in reference 3 on 1000- and 3000-hour aged T-111. The edge cracking observed in two of the three tube specimens could be due to differences in sample preparation. Incomplete removal of worked metal in some specimens could cause cracking in the 100-hour aged tubing. Chemical analyses of the two tube specimens that had edge cracks indicated insignificant increases in hydrogen and oxygen content compared with the as-received and 1316° C annealed specimens.

Photographs following the ductility tests of the tube and sheet specimens in the as-received, 1316° C annealed, and 1040° C aged conditions are shown in figures 3 to 5. The as-received tube and sheet specimens were free of precipitates. Specimens vacuum annealed at 1316° C for 2 hours showed some grain-boundary precipitates. Specimens aged in vacuum at 1040° C for 100 hours had considerable intergranular precipitation. Aged tube specimens contained more precipitates than the aged sheet specimens. Precipitates recovered from 1040° C aged T-111 have been identified as hafnium dioxide (HfO_2) (ref. 3). There is no apparent reason why the aged tubing contained more precipitates than the aged sheet, since the hafnium content of each was similar (table II). The presence of these particles near the surface did not cause embrittlement.

No Lithium Removal

Ductility tests were made on both tube and sheet specimens exposed to lithium for 100 hours at 1040° C and with lithium still remaining on the surface to determine if there was embrittlement resulting from the heat treatment in lithium. The tests were conducted in dry argon immediately after opening the capsules. All of these specimens were ductile, indicating that heat treatment in lithium did not affect the ductility. Similar tests were also conducted on specimens that were annealed and then wet with lithium. There was no apparent ductility difference between unaged or 100-hour, 1040° C aged specimens. Chemical analysis of these specimens was not possible since the determinations cannot be made on lithium coated specimens.

Liquid Ammonia Cleaning Treatment

Tube and sheet specimens that had been annealed and then wet with lithium or aged at 1040° C in lithium were cleaned by rinsing in liquid ammonia. Specimens that had been vacuum annealed at 1316° C or vacuum aged at 1040° C were also rinsed in liquid ammonia to determine if this cleaning technique affected the T-111 not exposed to lithium.

All sheet specimens cleaned by liquid ammonia rinsing were ductile on subsequent bending in air or in dry argon. Those ring specimens annealed, those annealed and wet with lithium, and those vacuum aged at 1040° C were also ductile upon bending in dry argon. (No tests were conducted on those specimens in air.) The one ring specimen aged at 1040° C with lithium and flattened in air developed edge cracks; the two other ring specimens flattened in dry argon were ductile. Again, the edge cracking observed on the aged T-111 tube specimens could be due to surface preparation.

Following lithium removal, specimens from each heat treatment were exposed to moist air for 1 week. The purpose of this exposure was to determine if lithium removal was complete. If removal was incomplete, then exposure to moist air would tend to cause hydrogen embrittlement by reaction of the moisture with lithium. Specimens annealed and wet with lithium and specimens aged at 1040° C in lithium were both ductile following moist air exposure. Chemical analyses showed no significant changes in the interstitial concentrations following the moist air exposure. It is apparent that liquid ammonia removes lithium adequately from T-111.

Micrographs of tube and sheet specimens aged at 1040° C with lithium followed by liquid ammonia cleaning and moist air exposure are shown in figure 6. Both of these specimens resemble those heat treated at 1040° C without lithium. The intergranular precipitates do not appear to be affected by lithium exposure. Tube specimens show more subsurface precipitates than do sheet specimens, similar to the specimens aged at 1040° C without lithium (fig. 5).

Vacuum Distillation Cleaning Treatment

Tube and sheet specimens that had been annealed at 1316° C and wet with lithium and specimens aged at 1040° C in lithium were cleaned by the vacuum distillation method. Similar specimens not exposed to lithium were also given the vacuum distillation treatment to determine if they were affected by heating to 700° C.

All sheet specimens cleaned by vacuum distillation were ductile on bending in air and in dry argon. There was no ductility loss on any of the as-received or vacuum annealed tube specimens regardless of bending atmosphere, indicating that T-111 not exposed to lithium was unaffected by vacuum distillation conditions. All the tube specimens annealed and wet with lithium or aged with lithium were ductile regardless of test atmosphere. One ring specimen from the tube aged with lithium at 1040° C for 100 hours was ductile when flattened in the dry box. The second specimen developed edge cracks during flattening in air. The tube specimens that were vacuum aged at 1040° C and flattened in dry argon also had edge cracks. (No tests of the vacuum aged material were conducted in air.) Again, edge cracking was observed only on the 100-hour aged T-111 tube specimens and appears to be independent of the test atmosphere and exposure to lithium. It is possible that the aged T-111 is more sensitive to the presence of worked metal, and, because of inconsistencies in surface preparation, some specimens developed edge cracks during testing. Chemical analysis of the specimens aged at 1040° C in lithium was similar to those for specimens annealed at 1316° C and wet with lithium and specimens vacuum aged at 1040° C.

Following the vacuum distillation treatment, two tube and two sheet specimens that had been aged at 1040° C in lithium were exposed to moist air for 1 week before testing. All of the samples, both tube and sheet, were ductile. There was no change in interstitial content following moist air exposure. The microstructures of tube and sheet specimens, which had been aged at 1040° C in lithium, cleaned by vacuum distillation, and exposed to moist air, are similar to those shown previously in figures 5 and 6.

From these results the vacuum distillation technique appears to be adequate to remove lithium from 1040° C aged T-111 without causing embrittlement or interstitial contamination. Heating of the T-111 to 700° C at 1.3×10^{-3} newton per square meter (1×10^{-5} torr) or less for several hours does not appear to alter the sample. Chemical and metallographic results obtained on specimens cleaned by the distillation treatment are similar to those obtained on samples cleaned by the low-temperature liquid ammonia treatment. The results obtained on samples heat treated with lithium and cleaned by either vacuum distillation or liquid ammonia rinsing were similar to those obtained on specimens heat treated without lithium.

Moist Air Exposure Treatment

Sheet specimens that had been aged at 1040° C with and without lithium were exposed to moist air for 1 week. The single sheet specimen not exposed to lithium was ductile when tested in air or dry argon. Two sheet specimens exposed to lithium had lithium adhering to the surface. One specimen had a heavy lithium coating (about 0.15 centimeter (1/16 in.) thick) on each side; the other had only a thin film on each side. The lithium was completely oxidized on both samples after the 1-week moist air exposure. These samples were rinsed with water to remove lithium hydroxide, dried, and tested. The first specimen (fig. 7(a)) bent completely, but developed a crack nearly through the material. Cracking was intergranular and grain separation occurred near the crack. The other specimen (fig. 7(b)), however, was ductile. Analysis of the first sample indicated that the hydrogen content increased to 6 ppm, apparently causing embrittlement. Analysis of the second (ductile) specimen indicated no change in interstitial element content following the moist air exposure. Hydrogen pickup by the first sample was probably due to the heavier lithium coating.

Uncleaned tube specimens that had been aged at 1040° C with and without lithium and that had been annealed and wet with lithium were exposed to moist air for 1 week. The specimens were then rinsed with water, dried, and evaluated. The specimen annealed and wet with lithium was ductile in air. The specimen aged at 1040° C in vacuum developed edge cracks on bending in dry argon. Of three specimens aged at 1040° C in lithium, two were ductile and the third developed edge cracks on bending in air. Chemical analysis of the tubing specimens showed no hydrogen pickup, which is consistent with the observation of good ductility. Microstructure of these tube specimens resembled those of other specimens with the same thermal history.

The results of the moist air exposure indicate the unpredictable nature of hydrogen embrittlement. One would expect the lithium coated specimens to be brittle when exposed to moist air conditions (ref. 4). From these results, it is apparent that 100-hour aged T-111 may not be as sensitive to hydrogen embrittlement as material aged for longer times.

Water Removal Treatment

One tubing specimen and two sheet specimens that had been annealed and wet with lithium were submerged in water for about 30 minutes to remove the residual lithium. The tubing specimen shown in figure 8(a) was flattened in the argon atmosphere, and it cracked almost completely across the wall. The cracking was intergranular. Both sheet specimens, one bent in air and the other in dry argon, fractured after bending about 20°. One is shown in figure 8(b). Chemical analyses of the sheet and tube specimens showed hydrogen concentrations of 142 and 24 ppm, respectively. There also appeared to be a slight oxygen increase in both specimens. Cracking in the sheet specimens was mainly intergranular; however, some transgranular cracking, possibly due to a locally high hydrogen content, is evident at the surface.

CONCLUSIONS

The following conclusions were drawn from this study of lithium removal techniques for T-111.

1. The liquid ammonia and vacuum distillation techniques remove trace lithium from 1040° C aged T-111 completely and thus do not allow residual lithium reaction with atmospheric moisture, which could lead to contamination and embrittlement.

2. There is no apparent difference between the liquid ammonia and vacuum distillation techniques for removal of lithium from T-111.

3. As described in this and other reports, hydrogen contamination and embrittlement can develop in T-111 from cleaning processes that liberate hydrogen.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 30, 1973,
503-25.

REFERENCES

1. Harrison, R. W.; and Smith, J. P.: Advanced Refractory Alloy Corrosion Loop Program. Rep. GESP-562, General Electric Co. (NASA CR-72818), Nov. 12, 1970.

2. Watson, Gordon K.: Preliminary Evaluation of T-111 Clad UN Fuel Specimens from 2500-Hour 1040° C (1900° F) Lithium Loop Test. NASA TM X-52998, 1971.

3. Watson, Gordon K.; and Stephens, Joseph R.: Effect of Aging at 1040° C (1900° F) on the Ductility and Structure of a Tantalum Alloy, T-111. NASA TN D-6988, 1972.

4. Reeves, J. D.; and Hand, R. B.: The Effects of Lithium Stripping from Cb-1Zr and T-111 Test Specimens Using Water, Ethanol, Butyl Cellosolve or Liquid Ammonia as a Solvent. Rep. GESP-471, General Electric Co., June 10, 1971.

5. Sessions, C. E.; and De Van, J. H.: Effect of Oxygen, Heat Treatment, and Test Temperature on the Compatability of Several Advanced Refractory Alloys with Lithium. Rep. ORNL-4430, Oak Ridge National Lab., Apr. 1971.

TABLE I. - DUCTILITY RESULTS[a] OF T-111 TUBE AND SHEET SPECIMENS SUBJECTED TO VARIOUS HEAT TREATMENTS AND LITHIUM REMOVAL CONDITIONS

Lithium removal condition	Heat treatment									
	As-received, no lithium		Annealed,[b] no lithium		Annealed,[b] lithium		1040° C - 100 hr, no lithium		1040° C - 100 hr, lithium	
	Tube	Sheet	Tube	Sheet	Tube	Sheet	Tube	Sheet	Tube	Sheet
Lithium absent, no removal treatment	D^c D^c -----	D^c --	D^c --	D^c --	------ ------	----- -----	EC EC D^c ---------	D D^c D D	-------- --------	------ ------
No lithium removal	-----	--	--	--	D	D	---------	----	D D	D
Liquid ammonia treatment	-----	--	D	D	D	D D^c	D	D D	D D EC^c	D D^c
Liquid ammonia treatment followed by moist air exposure	-----	--	--	--	D D^c	D D^c	---------	----	D	D D D^c
Vacuum distillation treatment	----- -----	-- --	D^c --	D^c --	D D D^c ------	D D^c D D^c	EC ---------	D D ----	D EC^c --------	D D^c D D^c
Vacuum distillation treatment followed by moist air exposure	----- -----	-- --	-- --	-- --	D D ------	D D^c D D^c	--------- ---------	---- ----	D D --------	D D^c D D^c
Moist air exposure without lithium removal treatment	----- -----	-- --	-- --	-- --	D^c ------	----- -----	EC ---------	D D^c ----	D D^c EC^c D^c	B^c B D^c D^c
Water removal	----- -----	-- --	-- --	-- --	B ------	B B B^c B^c	--------- ---------	---- ----	-------- --------	------ ------

[a]D, ductile; B, brittle; EC, edge cracks.
[b]Annealed conditions: 1316° C for 2 hr.
[c]Ductility test done in air; others done in argon atmosphere.

TABLE II. - CHEMICAL ANALYSES FOR INTERSTITIAL ELEMENTS IN T-111 TUBE[a] AND SHEET[b] SPECIMENS SUBJECTED TO VARIOUS HEAT TREATMENTS AND LITHIUM REMOVAL CONDITIONS

Lithium removal condition	Heat treatment																													
	As-received, no lithium						Annealed, no lithium						Annealed, lithium						1040° C - 100 hr, no lithium						1040° C - 100 hr, lithium					
	Tube			Sheet			Tube			Sheet			Tube			Sheet			Tube			Sheet			Tube			Sheet		
	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H
Lithium absent, no removal treatment	40	13	1.1	171	10	<0.5	26	13	<0.5	174	9	<0.5	--	--	---	---	--	----	38	15	1.9	162	13	<0.5	--	--	---	---	--	----
	--	--	---	168	11	<.5	--	--	----	---	-	----	--	--	---	---	--	----	35	14	1.6	---	--	----	--	--	---	---	--	----
Liquid ammonia treatment	--	--	---	---	--	----	--	--	----	---	-	----	33	15	1.0	170	12	<0.5	36	16	1.7	171	10	.86	35	16	2.2	132	10	<0.5
Liquid ammonia treatment followed by moist air exposure	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	----	--	--	---	---	--	----	31	16	2.1	134	10	.63
Vacuum distillation treatment	--	--	---	---	--	----	--	--	----	---	-	----	35	13	1.7	178	12	1.3	36	15	1.5	162	10	.63	32	14	1.0	157	15	2.4
Vacuum distillation treatment followed by moist air exposure	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	---	--	--	---	---	--	----	31	11	1.1	148	15	2.3
	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	---	--	--	---	---	--	----	--	--	---	133	11	.98
Moist air exposure without lithium removal treatment	--	--	---	---	--	----	--	--	----	---	-	----	46	13	1.0	---	--	---	--	--	---	---	--	----	38	16	2.2	155	13	6.1
	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	---	--	--	---	---	--	----	--	--		136	12	.94
Water removal	--	--	---	---	--	----	--	--	----	---	-	----	52	16	24.0	184	15	142	--	--	---	---	--	----	--	--	---	---	--	----

[a]Contained 7.9 percent tungsten and 1.9 percent hafnium.
[b]Contained 8.5 percent tungsten and 2.0 percent hafnium.

TABLE II. - CHEMICAL ANALYSES FOR INTERSTITIAL ELEMENTS IN T-111 TUBE[a] AND SHEET[b] SPECIMENS

SUBJECTED TO VARIOUS HEAT TREATMENTS AND LITHIUM REMOVAL CONDITIONS

Lithium removal condition	Heat treatment																													
	As-received, no lithium						Annealed, no lithium						Annealed, lithium						1040° C - 100 hr, no lithium						1040° C - 100 hr, lithium					
	Tube			Sheet			Tube			Sheet			Tube			Sheet			Tube			Sheet			Tube			Sheet		
	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H	O	N	H
Lithium absent, no removal treatment	40	13	1.1	171	10	<0.5	26	13	<0.5	174	9	<0.5	--	--	---	---	--	----	38	15	1.9	162	13	<0.5	--	--	---	---	--	----
	--	--	---	168	11	<.5	--	--	----	---	-	----	--	--	---	---	--	----	35	14	1.6	---	--	----	--	--	---	---	--	----
Liquid ammonia treatment	--	--	---	---	--	----	--	--	----	---	-	----	33	15	1.0	170	12	<0.5	36	16	1.7	171	10	.86	35	16	2.2	132	10	<0.5
Liquid ammonia treatment followed by moist air exposure	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	----	--	--	---	---	--	----	31	16	2.1	134	10	.63
Vacuum distillation treatment	--	--	---	---	--	----	--	--	----	---	-	----	35	13	1.7	178	12	1.3	36	15	1.5	162	10	.63	32	14	1.0	157	15	2.4
Vacuum distillation treatment followed	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	---	--	--	---	---	--	----	31	11	1.1	148	15	2.3
by moist air exposure	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	---	--	--	---	---	--	----	--	--	---	133	11	.98
Moist air exposure without lithium	--	--	---	---	--	----	--	--	----	---	-	----	46	13	1.0	---	--	---	--	--	---	---	--	----	38	16	2.2	155	13	6.1
removal treatment	--	--	---	---	--	----	--	--	----	---	-	----	--	--	---	---	--	---	--	--	---	---	--	----	--	--		136	12	.94
Water removal	--	--	---	---	--	----	--	--	----	---	-	----	52	16	24.0	184	15	142	--	--	---	---	--	----	--	--	---	---	--	----

[a]Contained 7.9 percent tungsten and 1.9 percent hafnium.
[b]Contained 8.5 percent tungsten and 2.0 percent hafnium.

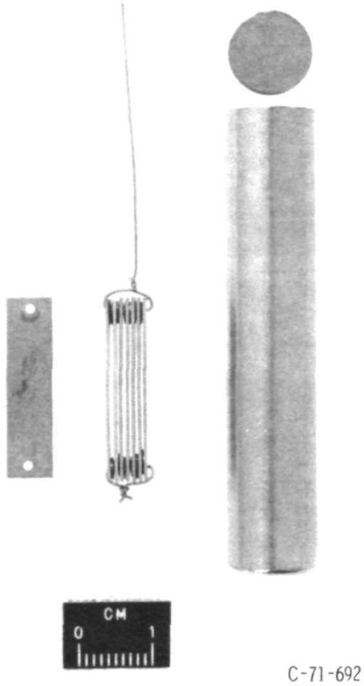


Figure 1. - T-111 capsule and sheet specimens.

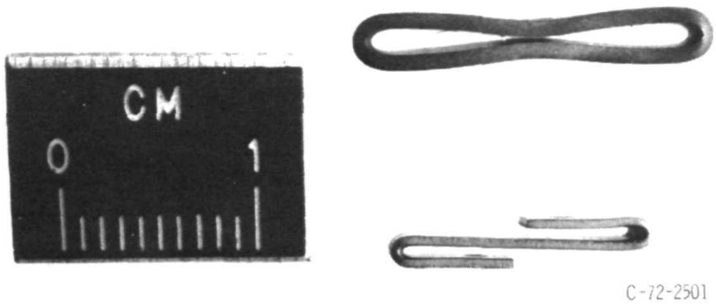
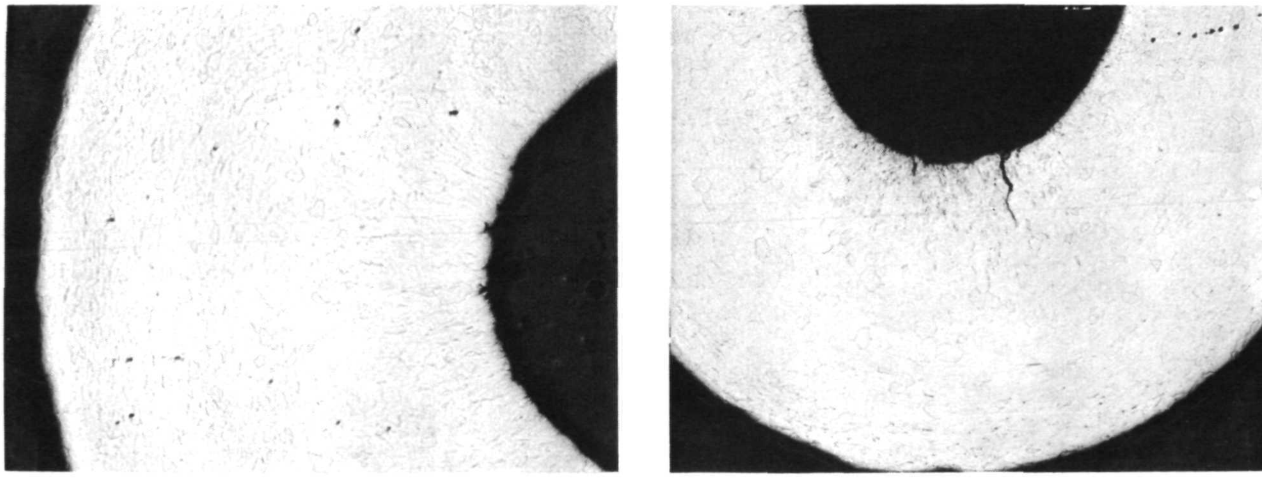


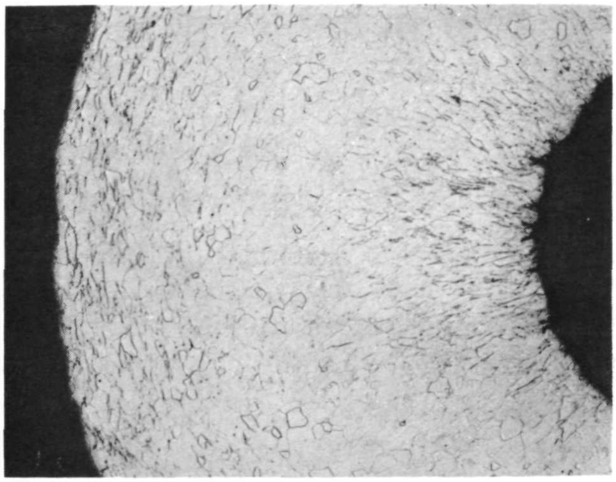
Figure 2. - T-111 tube and sheet specimens following ductility testing.



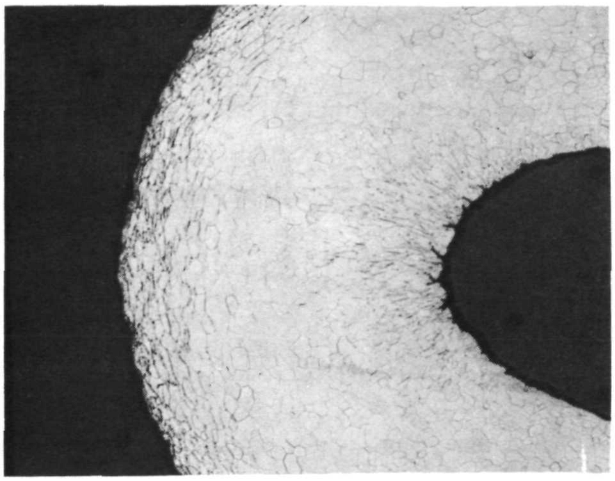
(a) Tubing specimen. Oxygen, 40 ppm; hydrogen, 1.1 ppm; nitrogen, 13 ppm.

(b) Sheet specimen. Oxygen, 168 ppm; hydrogen, 0.5 ppm; nitrogen, 11 ppm.

Figure 3. - As-received T-111 after room temperature ductility test in air. Etchant: 30 grams ammonium bifluoride, 50 cubic centimeters nitric acid, and 20 cubic centimeters water. X100.

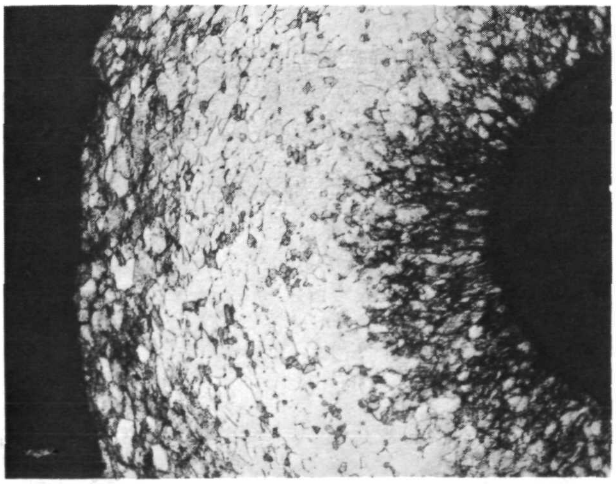


(a) Tubing specimen. Oxygen, 26 ppm; hydrogen, 0.5 ppm; nitrogen, 13 ppm.

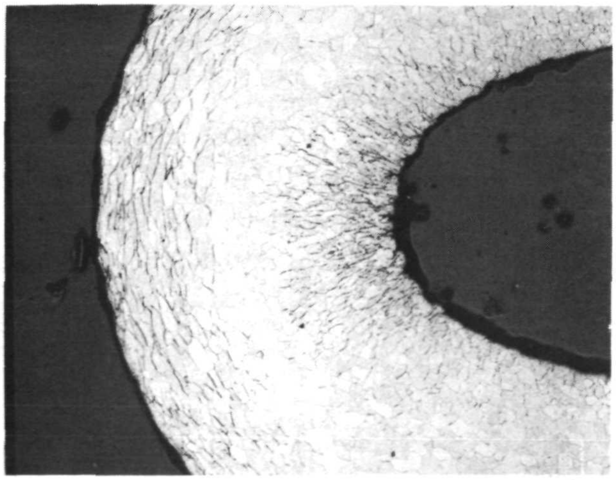


(b) Sheet speciemn. Oxygen, 174 ppm; hydrogen, 0.5 ppm; nitrogen, 9 ppm.

Figure 4. - T-111 annealed for 2 hours at 1316^{0} C. Ductility test performed in air. Etchant: ammonium bifluoride, nitric acid, and water. X100.

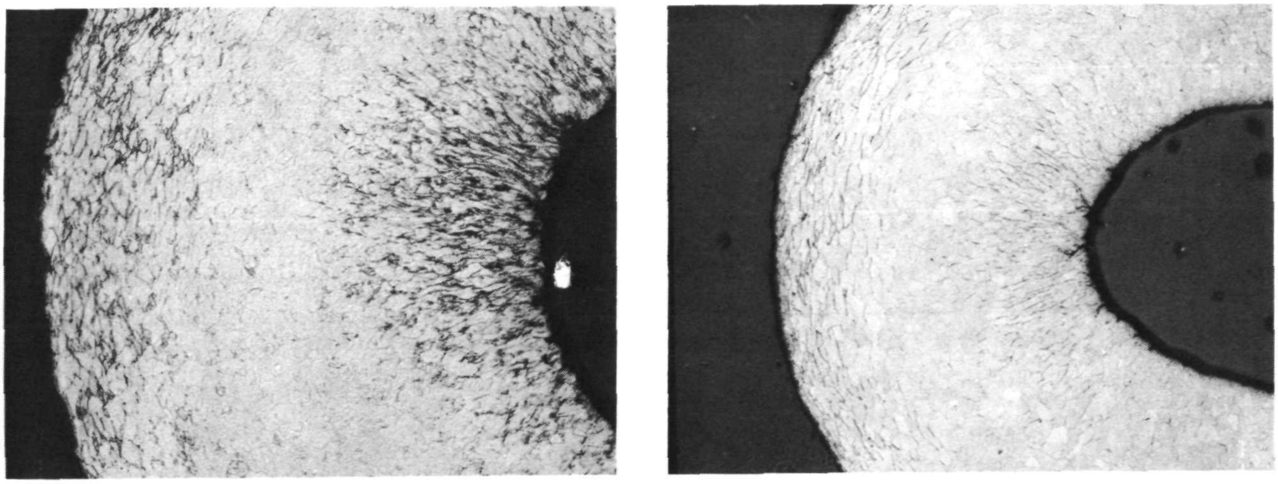


(a) Tubing specimen. Oxygen, 35 ppm; hydrogen, 1.6 ppm; nitrogen, 14 ppm.



(b) Sheet specimen. Oxygen, 162 ppm; hydrogen, 0.5 ppm; nitrogen, 13 ppm.

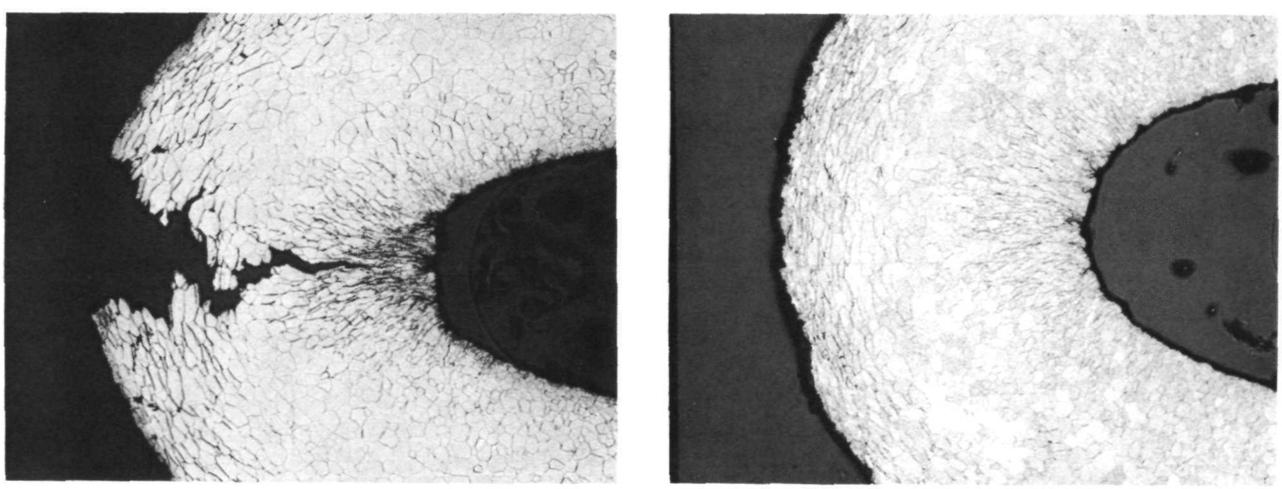
Figure 5. - T-111 aged at 1040^{0} C for 100 hours. Ductility tests performed in argon atmosphere. Etchant: ammonium bifluoride, nitric acid, and water. X100.



(a) Tubing specimen. Oxygen, 31 ppm; hydrogen, 2.1 ppm; nitrogen, 16 ppm.

(b) Sheet specimen. Oxygen, 134 ppm; hydrogen, 0.63 ppm; nitrogen, 10 ppm.

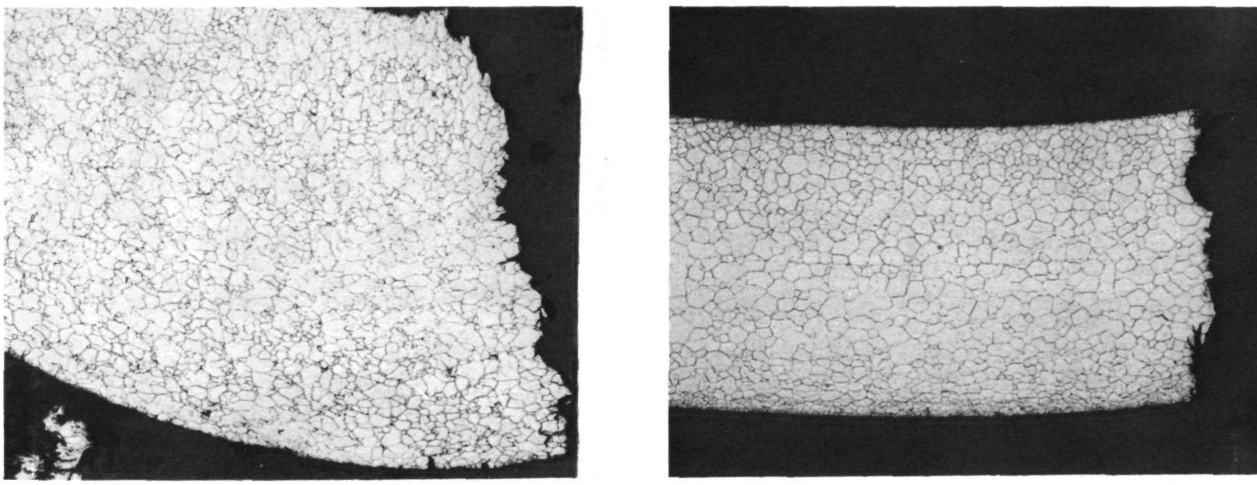
Figure 6. - T-111 exposed to lithium for 100 hours at 1040° C. Specimens cleaned with liquid ammonia and then exposed to moist air. Ductility tests performed in argon atmosphere. Etchant: ammonium bifluoride, nitric acid, and water. X100.



(a) Specimen brittle. Oxygen, 155 ppm; hydrogen, 6.1 ppm; nitrogen, 13 ppm.

(b) Specimen ductile. Oxygen, 136 ppm; hydrogen, 0.94; nitrogen, 12 ppm.

Figure 7. - T-111 sheet specimens exposed to lithium for 100 hours at 1040° C. Specimens exposed to moist air. Ductility tests performed in ambient air. Etchant: ammonium bifluoride, nitric acid, and water. X100.



(a) Tubing specimen. Oxygen, 52 ppm; hydrogen, 24.0 ppm; nitrogen, 16 ppm.

(b) Sheet specimen. Oxygen, 184 ppm; hydrogen, 142 ppm; nitrogen, 15 ppm.

Figure 8. - T-111 specimens annealed and exposed to lithium. Lithium removed with water. Ductility tests performed in argon atmosphere. Both specimens brittle. Etchant: ammonium bifluoride, nitric acid, and water. X100.

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