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EFFECT OF AGING AT 1040° C (1900° F) ON THE DUCTILITY AND STRUCTURE OF A TANTALUM ALLOY, T-111

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SUMMARY

This study was conducted to investigate the observed embrittlement of T-111 (a tantalum alloy containing 8 percent tungsten and 2 percent hafnium) following long time aging at 1040° C (1900° F). The main objectives were to determine the probable cause or causes of the post-aging embrittlement and possible means of avoiding it. Samples of T-111 sheet and tubing were aged for up to about 3000 hours at 1040° C (1900° F) in either vacuum or liquid lithium. These samples were evaluated primarily on the basis of ductility and were compared with results on unaged T-111. An extensive metallographic study also was conducted on aged T-111 samples from this study and from other programs.

The test results indicate that the brittle, intergranular fractures observed in aged T-111 were caused by hydrogen embrittlement during post-aging handling operations. Aging the T-111 at 1040° C (1900° F) for long periods of time greatly increased the sensitivity of the T-111 to hydrogen embrittlement. Hydrogen pickup, with the resulting embrittlement, occurred by exposure of the T-111 to water during cutting or sanding operations after aging. Testing of aged T-111 tubing specimens in a moist atmosphere also resulted in embrittlement. This type of embrittlement in T-111 apparently can be avoided by preventing exposure of thermally aged T-111 to moist atmospheres during post-aging handling operations. Vacuum annealing of the aged and embrittled T-111 for 1 hour at 1040° C (1900° F) restored the ductility.

Metallographic studies showed that T-111 aged at 1040° C (1900° F) contained numerous hafnium dioxide (HfO_2) particles located primarily at grain boundaries. In contrast, the as-received material and samples aged at 1200° to 1315° C (2200° to 2400° F) were essentially free of precipitate particles. Bend tests on T-111 aged at 1040° C (1900° F) resulted in edge and surface cracking at -196° C (-321° F). Samples were tested without any surface preparation after aging. Ductile behavior was observed for 1315° C (2400° F) aged T-111 when tested under similar conditions. These results suggest that precipitation of HfO_2 during aging at 1040° C (1900° F) is responsible for the loss of ductility at -196° C (-321° F) of aged T-111 and may account for the increased sensitivity of aged T-111 to hydrogen embrittlement.

INTRODUCTION

Various refractory metals are being investigated for space power applications. A tantalum alloy, T-111 (Ta-8W-2Hf), has been selected (ref. 1) as the reference alloy for many high-temperature space power systems because of its good fabricability, its relatively good high-temperature strength, and its resistance to alkali metal corrosion. Although the T-111 alloy appears to have very good ductility, occasional cracking of the alloy has been observed during processing and fabrication (ref. 2). In some cases brittle, intergranular propagation of cracks was observed under applied stress at room temperature following cutting or machining operations.

The response of T-111 to long-time aging was studied in detail by Lessman and Gold (ref. 3). Some reduction in ductility occurred when gas-tungsten-arc welded samples of T-111 were aged at temperatures of 980° C (1800° F) to 1150° C (2100° F) for up to 10 000 hours. These aging treatments had no apparent effect, however, on the ductility of unwelded T-111.

As part of the evaluation of a T-111 Rankine System Corrosion Test Loop conducted by Harrison and Smith (ref. 4), ring shaped samples, cut from the tubing in various parts of the loop, were flattened at room temperature to determine ductility. These tests, conducted at the completion of the 10 000-hour loop test, showed a variation in sample ductility. Samples from the portion of the loop operated at about 1200° C (2200° F) were relatively ductile and could undergo considerable deformation with little or no cracking. Samples from the 1040° C (1900° F) part of the loop were very brittle and fractured in a completely intergranular manner with very little deformation.

Another T-111 pumped-loop test was conducted to investigate the chemical compatibility of T-111 clad, uranium mononitride (UN), simulated fuel element specimens with flowing lithium at 1040° C (1900° F). Two of the fuel-element specimens were removed from the loop after 2500 hours and evaluated on the basis of chemistry, metallography, and cladding ductility (ref. 5). Although very little change in interstitial content of the T-111 cladding was observed, room-temperature ductility tests on the cladding resulted in brittle, intergranular fracture as shown in figure 1. The pickup of trace amounts of hydrogen was proposed as a possible cause of the embrittlement because the ductility of the T-111 could be restored by vacuum annealing for one hour at 1315° C (2400° F).

Because of the lack of room-temperature ductility seen in some of the aged T-111 samples, a program was initiated to investigate experimentally some of the factors that could contribute to the observed loss of ductility. The studies described in this report are part of this program. The major objectives of these studies were to determine the nature and probable cause or causes of the post-aging embrittlement of T-111 and to identify ways of avoiding the embrittlement. Toward these objectives, various aged T-111 specimens were examined primarily by ductility tests and metallography. The effects of

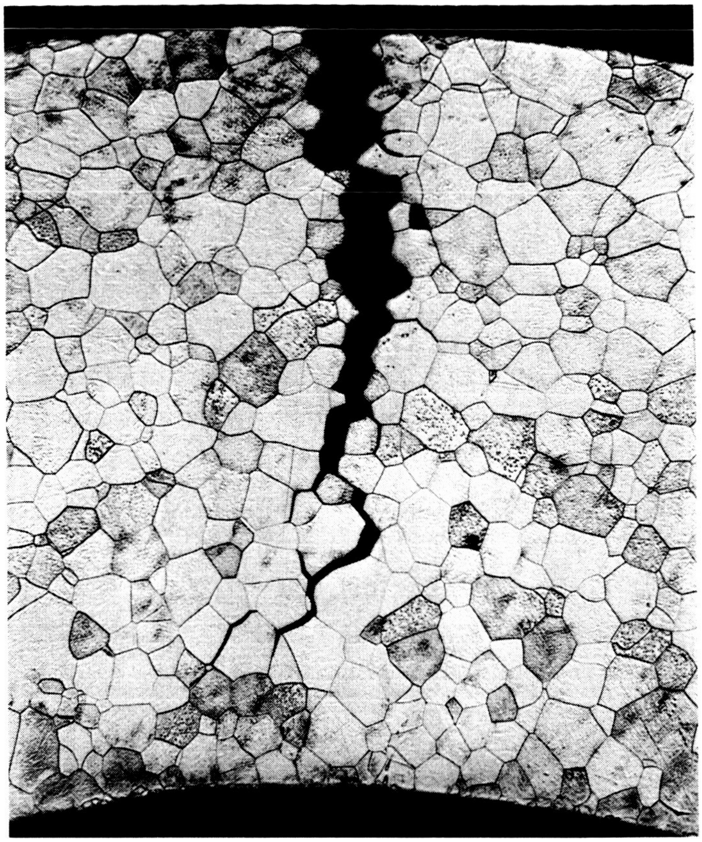


Figure 1. - Intergranular fracture in ring ductility test on T-111 cladding from fuel-element specimen after 2500 hours in 1040° C (1900° F) lithium loop. Etchant, 30 grams ammonium bifluoride, 50 milliliters nitric acid, and 20 milliliters water.

the thermal aging variables of temperature, time, and atmosphere were studied along with various methods of post-aging handling operations.

PROCEDURE

Sample History

Most of the ductility studies described in this report were conducted on T-111 tubing and sheet samples aged specifically for this study. The metallographic study was conducted on samples from this ductility study and on representative samples from several other investigations. The histories of the various samples used in this report are described in the following two sections.

Samples for ductility tests. - Commercially produced T-111 tubing and sheet samples were aged for up to 3000 hours in an ultrahigh vacuum (1.3×10^{-7} N/m^2; 1×10^{-9} torr) furnace at 1040° C (1900° F), the temperature where most of the previously observed aging embrittlement occurred. To determine what effect a liquid alkali metal would have on the aged samples, some of the samples were in contact with lithium during aging. Two heats of T-111 sheet and one heat of T-111 tubing were studied.

The sheet samples were encapsulated in the tubing samples for the aging heat treatments. Six test capsules were fabricated from T-111 tubing having a 1.27-centimeter (0.500-in.) outside diameter and a wall thickness of 0.081 centimeter (0.032 in.). The sheet samples were sheared from the two lots of 0.051-centimeter (0.020-in.) thick T-111 sheet. Sheet edges were dressed by wet sanding with 400-grit silicon carbide paper. Before assembly, all the T-111 components were chemically cleaned using the techniques described in reference 6 and then annealed for 1 hour at 1090° C (2000° F) in a vacuum of 1.3×10^{-3} newtons per square meter (1×10^{-5} torr) or better. The capsules were sealed by electron beam welding T-111 end caps in place in a vacuum of 1.3×10^{-2} newtons per square meter (1×10^{-4} torr) or better.

Three of the test capsules contained lithium; the remaining three did not. Aging times and specimen details are summarized in table I.

TABLE I. - SUMMARY OF T-111 CAPSULES USED IN DUCTILITY STUDY. ALL CAPSULES AGED AT 1040° C (1900° F)

Capsule number	Aging time, hr	Contact with lithium	Number of sheet specimens	Sheet heat number
1	0	No	7	650085
2	0	Yes	4	650085
3	1128	No	7	650085
4	1128	Yes	4	650085
5	2862	No	7	650087
6	2862	Yes	4	650087

After aging, the lithium was removed from the three lithium-filled capsules by vacuum distillation. Samples of both the aged sheet and the aged tubing were evaluated on the basis of ductility and sensitivity of the samples to various preparation and testing techniques. The results on the aged samples were compared with the results obtained on as-received, unaged samples.

Samples for metallographic studies. - In addition to the metallographic examination of the samples used in the ductility tests, an extensive metallographic study was conducted on aged T-111 tubing and sheet samples from other NASA-sponsored programs.

Tubing samples were cut from a Rankine system corrosion test loop (ref. 4) after completion of 10 000 hours of operation. During operation, the T-111 tubing contained potassium vapor while the outside diameter was exposed to a vacuum of 1.3×10^{-7} newtons per square meter (1×10^{-9} torr). Specimens for metallographic examination were cut from the 1040° C (1900° F) portion and the 1200° C (2200° F) portion of the loop. Also, samples of T-111 sheet for metallographic examination were obtained from a Lewis program that involved investigation of the effects of aging in lithium on the mechanical properties of T-111. These sheet samples were from one of the same lots (Heat 650087) of material used in the ductility tests. They were exposed to lithium or a vacuum of 1.3×10^{-7} newtons per square meter (1×10^{-9} torr) for up to 5000 hours over the temperature range 980° C (1800° F) to 1315° C (2400° F).

Ductility Tests

The ductility of the T-111 tubing was evaluated using rings cut from each of the T-111 capsules. The various procedures used for preparing the samples for the ductility test are described in the RESULTS section of this report. These rings were subjected to a simple flattening test in a vise at room temperature. No attempt was made on these tests to control strain rate or test environment. Each ring specimen was flattened until the two opposing inside surfaces of the ring contacted one another or until the specimen fractured.

The bend ductility was determined for the T-111 sheet samples from each of the test capsules. The test temperature ranged from room temperature (24° C; 75° F) to liquid nitrogen temperature (-196° C; -321° F). All tests were conducted in a screw-driven tensile-testing machine at a punch rate of 2.54 centimeters per minute (1 in./min.). A bend radius of 1t (0.051 cm, 0.020 in.) and a total bend angle of about 160° was used for all the tests. The room-temperature test specimens were flattened in a vise after completing the 1t bend in the tensile machine. So, in effect, the room temperature tests were a 180°, 0t bend.

Metallographic Techniques

The metallographic characterization included the use of standard light microscope techniques, transmission electron microscopy, and scanning electron microscopy plus particle identification using X-ray diffraction, electron microprobe, and characteristic X-ray analysis on the scanning electron microscope.

Standard metallographic techniques were used to prepare specimens for light microscope observations. Polished specimens were etched with either a solution of three parts glycerine, two parts nitric acid, and one part hydrofluoric acid or a solution of 20 milliliters water, 50 millilileters nitric acid, and 30 grams of ammonium bifluoride.

Thin foils for observation in the electron microscope were prepared from disks cut from T-111 tubing and sheet using electrical discharge machining. Thinning was accomplished using a solution of 85 percent sulfuric acid and 15 percent hydrofluoric acid, which was near room temperature during electropolishing. A current density of approximately 30 milliamperes per square centimeter resulted in a highly polished surface.

Extracted precipitate particles from bulk T-111 samples were obtained by dissolving the matrix in a solution containing 90 milliliters methanol, 10 milliliters bromine, and 10 grams of tartaric acid. The samples were supported in the solution on a platinum screen during dissolution. After dissolution, repeated centrifuging and methanol rinsing were performed to clean the undissolved residue. Particles were analyzed by X-ray diffraction using the standard Debye-Scherrer technique.

RESULTS

Chemical Analysis

The complete vendor's analyses for the T-111 tubing and for the two lots of T-111 sheet are presented in table II. Additional analyses for interstitials in the T-111 tubing and sheet, both before and after aging, are listed in table III. For comparison a series of samples (designated 0 time in table III) underwent the capsule loading operation with and without lithium, but were not subsequently aged at 1040° C (1900° F). Some differences can be noted between the vendor's analyses and our analyses. The vendor indicated that heat 650087 contained more oxygen than heat 650085; our analyses indicates that the reverse was true. In addition, the carbon contents reported by the vendor for both heats of sheet material are lower than those obtained by our analyses.

The only significant effect that can be seen as a result of aging is that the oxygen content of the sheet samples aged in lithium decreased markedly. All of the other interstitials in the T-111 sheet samples were essentially unchanged. Aging had no apparent effect on the interstitial content of the tubing samples.

TABLE II. - VENDOR'S ANALYSIS OF T-111

USED IN STUDY

[Values are in ppm by weight except as noted.]

Element	Tubing Heat 8227	Sheet Heat 650085	Sheet Heat 650087
W	7.44 percent	8.5 percent	8.1 percent
Hf	2.30 percent	2.0 percent	1.8 percent
Ta	Balance	Balance	Balance
C	31	<50	50
O	26	120	160
N	18	15	20
H	5	2.9	1.9
Cb	35	470	450
Co	<5	<5	<5
Cr	<1	<10	<10
Cu	---	<40	<20
Fe	10	<20	<20
Mo	20	<10	<10
Ni	---	<10	<10
V	2	<10	<10
Zr	400	620	515

TABLE III. - EFFECT OF AGING ON THE INTERSTITIAL CONTENT

OF T-111 TUBING AND SHEET SPECIMENS

Aging condition		Element[a], ppm by weight			
Time at 1040° C (1900° F), hr	Contact with lithium	Carbon	Oxygen	Nitrogen	Hydrogen
Tubing					
0	No	33	57	24	<.5
0	Yes	28	57	26	<.5
1128	No	39	70	21	<.5
1128	Yes	37	40	22	<.5
2862	No	26	57	23	<.5
2862	Yes	36	58	25	.5
Sheet - heat 650085					
0	No	94	186	12	1.7
0	Yes	107	237	11	<.5
1128	No	111	235	11	<.5
1128	Yes	101	96	11	<.5
Sheet - heat 650087					
0	No	81	124	9	1.9
2862	No	84	120	7	<.5
2862	Yes	70	37	11	.6

[a]Carbon content determined by combustion. Oxygen, nitrogen, and hydrogen determined by vacuum fusion.

Ductility of Tubing Specimens

Effect of post-aging annealing. - Ring shaped specimens, approximately 0.6 centimeter (0.25 in.) long, were cut from each of the T-111 capsules using a water-cooled silicon carbide cutoff saw. The cut edges of all the ring specimens were dressed by wet sanding through 400-grit silicon carbide paper. One group of rings was flattened in the as-cut condition. A second group of rings was flattened after annealing in a vacuum of 1.3×10^{-3} newtons per square meter (1×10^{-5} torr) or better for 1 hour at 1040° C (1900° F). The 1040° C (1900° F) annealing temperature was selected because the aged T-111 had been at this temperature for extended periods of time. Thus, one additional hour at 1040° C (1900° F) should have essentially no effect on the properties of the aged T-111. A third set of specimens was prepared by cutting longer sections of tubing from the various capsules and annealing these sections in a vacuum of 1.3×10^{-3} newtons per square meter (1×10^{-5} torr) or better for 1 hour at 1040° C (1900° F). Then ring specimens were cut from the center part of each of the annealed tubing sections. The purpose of this third group of tests was to determine what effect annealing before cutting would have on ductility. The results of all these tests are summarized in table IV. The results are for single tests at each condition. (Because of a shortage of material, no tests in the annealed-and-cut condition were conducted on the capsules containing lithium and aged 2862 hours. The material from this capsule was used for other tests.)

TABLE IV. - EFFECT OF CUTTING AND ANNEALING ON THE

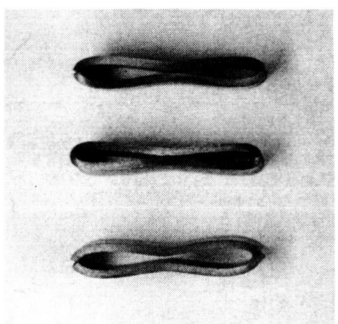
DUCTILITY OF UNAGED AND AGED T-111 TUBING[a]

Aging condition		Results of flattening tests		
Time at 1040° C (1900° F), hr	Contact with lithium	As-cut	Cut and annealed[c]	Annealed[c] and cut[b]
0	No	Ductile	Ductile	Ductile
	Yes	Ductile	Ductile	Ductile
1128	No	Edge cracks	Ductile	Edge cracks
	Yes	Edge cracks	Ductile	Edge cracks
2862	No	Edge and surface cracks	Ductile	Edge and surface cracks
	Yes	Fractured	Not tested	Not tested

[a]Ductility determined by tube flattening test at room temperature.
[b]Samples cut using a water-cooled, silicon carbide cutoff saw.
[c]Samples annealed at 1040° C (1900° F) for 1 hr in vacuum.

The flattened ring specimens from the capsule aged without lithium at 1040° C (1900° F) for 2862 hours are shown in figure 2. Very deep edge cracks can be seen in the rings tested in the as-cut condition and in the annealed-and-cut condition. No cracks were detected in the ring flattened in the annealed condition.

The results of these annealing studies indicate that the use of a water-cooled, silicon-carbide cutoff saw can cause embrittlement of T-111 that has been aged for long periods of time at 1040° C (1900° F). But this cutting method appeared to have no embrittling effect on unaged T-111. Since the ductility of the aged T-111 could be readily restored by a relatively low-temperature vacuum anneal, hydrogen pickup was suspected as the cause of the observed embrittlement. Hydrogen could have been picked-up in the cutting operation and removed with the subsequent vacuum anneal.


As-cut

Cut and annealed

Annealed and cut

Figure 2. - Effect of cutting or annealing on the ductility of T-111 tubing aged 2862 hours at 1040° C (1900° F) in vacuum.

Effect of specimen preparation technique. - Since the use of a water-cooled cutoff saw resulted in embrittlement of the aged T-111, other methods of preparing the specimens were investigated. A portion of the T-111 tubing aged without lithium for 2862 hours at 1040° C (1900° F) was cut into ring-shaped specimens using three different techniques: a water-cooled silicon carbide cutoff saw, a tubing cutter followed by filing, and a hacksaw (13 teeth/cm; 32 teeth/in.) followed by filing. The cut surfaces of all three rings were dressed by wet sanding through 400-grit silicon carbide paper. All three rings fractured in a brittle, intergranular manner during flattening at room temperature.

A fourth ring specimen (hacksawed, filed, and wet sanded) was annealed for 1 hour at 1040° C (1900° F) in a vacuum of 1.3×10^{-3} newtons per square meter (1×10^{-5} torr) or better. This ring was flattened completely at room temperature without any evidence of cracking. Again, the ductility was recovered with a relatively low-temperature annealing treatment, supporting the hypothesis of hydrogen embrittlement (ref. 5).

Although different methods were used to prepare the ring specimens for this series of tests, the final step in the preparation of all the samples was wet sanding of the cut surfaces. Thus, the observed embrittlement could have been caused by hydrogen picked up during wet sanding.

Finally, a fifth ring specimen was prepared by hacksawing, filing, and dry sanding rather than wet sanding. This ring also fractured upon flattening, but the amount of deformation that took place before fracture was greater than that observed for the wet sanded specimens.

Effect of ductility test atmosphere. - Because the aged T-111 ring specimen prepared without exposure to water fractured somewhat unexpectedly upon flattening in air, several sets of tests were conducted to determine if the test atmosphere could influence ductility. For the initial tests, three rings were prepared from a section of lithium-exposed T-111 tubing aged 2862 hours at 1040° C (1900° F). The rings were cut with a water-cooled silicon carbide cutoff saw. The cut surfaces were wet sanded through 400-grit silicon carbide paper. One ring was tested in ambient air; it fractured during flattening. The remaining two rings were tested in high purity argon. Both of these rings could be flattened completely without fracture; however, a few edge cracks were observed.

These tests indicated that the test atmosphere had a definite effect on the ductility of the ring samples. Some embrittlement of the specimens was expected because of the use of a water-cooled cutoff saw with the associated hydrogen pickup. The embrittlement seems to be concentrated near the cut surfaces as evidenced by the edge cracks that formed during the argon atmosphere tests. The edge cracks did not propagate during the tests in argon, but they did propagate during the tests in air.

Four more rings were cut from the same piece of tubing used in the previous tests (i.e., aged 2862 hours at 1040° C (1900° F) and containing lithium). The rings were cut with a hacksaw, filed, and dry sanded through 400-grit silicon carbide paper. Two rings were prepared in ambient air; the other two were prepared in argon. One ring of each type was flattened in either air or argon. The test results are summarized in table V. The ring specimens after flattening are shown in figure 3.

The test atmosphere had more influence on the ductility of the rings than did the preparation atmosphere. Both of the rings tested in air fractured; whereas, both rings tested in argon could be flattened completely with no evidence of cracking. No differences in ductility could be seen between the rings prepared in air and the rings prepared in argon.

The effect of test atmosphere also was determined on ring specimens cut from each of the capsules listed in table I. Two rings from each capsule were prepared in air by hacksawing, filing, and dry sanding. One ring from each capsule was flattened in ambient air, the other ring was flattened in argon. Test results are listed in table VI. The rings after testing are shown in figure 4.

All of the rings tested in argon were ductile. Of the rings tested in air, only the two rings in the unaged condition could be flattened without cracking. All of the aged T-111

TABLE V. - EFFECT OF SPECIMEN PREPARATION AND TEST ATMOSPHERES ON DUCTILITY OF T-111 TUBING CONTAINING LITHIUM AND AGED 2862 HOURS AT 1040° C (1900° F)

[All samples were prepared by hacksawing, filing, and dry-sanding through 400-grit silicon carbide paper.]

Preparation atmosphere	Ductility test atmosphere	Test results[a]
Air	Air	Fractured
Argon	Air	Fractured
Air	Argon	Ductile
Argon	Argon	Ductile

[a]Ductility determined by the flattening test of room temperature.

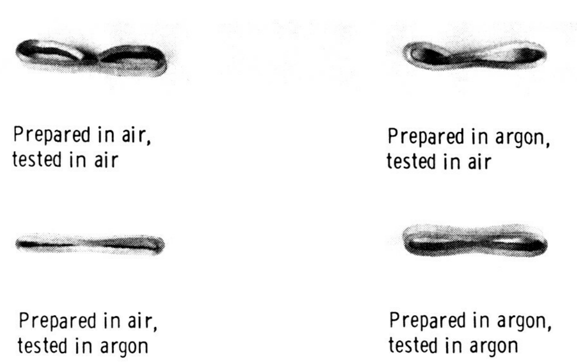


Figure 3. - Effect of preparation and test atmosphere on ductility of T-111 tubing containing lithium and aged 2862 hours at 1040° C (1900° F).

TABLE VI. - EFFECT OF TEST ATMOSPHERE ON

DUCTILITY OF UNAGED AND AGED T-111 TUBING

[All samples prepared in air by hacksawing, filing, and dry sanding.]

Aging condition		Test results[a]	
Hours at 1040° C (1900° F)	Contact with lithium	Ductility test atmosphere	
		Argon	Air
0	No	Ductile	Ductile
0	Yes	Ductile	Ductile
1128	No	Ductile	Edge crack
1128	Yes	Ductile	Fractured
2862	No	Ductile	Fractured
2862	Yes	Ductile	Fractured

[a]Ductility determined by tube flattening test at room temperature.

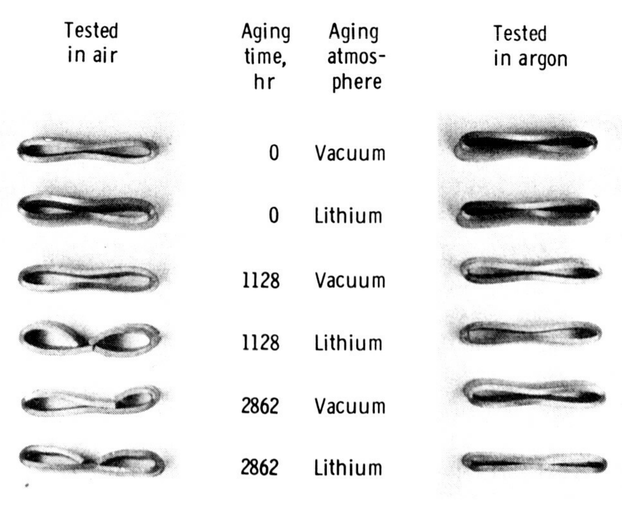


Figure 4. - Effect of test atmosphere on ductility of unaged and 1040° C (1900° F) aged T-111 tubing. All samples prepared in air by hack-sawing, filing, and dry sanding.

rings tested in air cracked to some extent upon flattening, with three of the four rings fracturing completely.

In an effort to identify the cause of the embrittlement observed when flattening aged T-111 in air, flattening tests were conducted in various atmospheres. Because of a shortage of the aged material used in the previous tests, the study on test atmospheres was performed on 1.9-centimeter (0.75-in.) diameter T-111 tubing having a 0.15-centimeter (0.060-in.) wall thickness. This tubing was aged in vacuum at 1040° C (1900° F) for 2862 hours at the same time as the six T-111 capsules described previously in this report. The ring samples were prepared by hacksawing, filing, and dry sanding.

The ring flattening tests were conducted in a sealed polyethylene bag filled with the desired atmosphere. The bag was purged with the gas being studied for about one-half hour before ring testing. Also before testing, the cut surfaces of the rings were sanded with 400-grit silicon carbide paper in the desired test atmosphere. This was done to remove any possible oxide layers on the cut surfaces that could minimize the reactions between the T-111 and the test atmosphere. The flattening results for the various test atmospheres are listed in table VII. The moist argon was produced by passing argon through a bubbler-bottle filled with water. No attempt was made to measure the moisture content of the argon. The tests in dry air were performed in a sealed, air-filled polyethylene bag containing anhydrous calcium sulfate crystals.

The ring specimen tested in ambient air and the one tested in moist argon both fractured in a brittle manner. The rings tested in all the other atmospheres could be flat-

TABLE VII. - EFFECT OF VARIOUS TEST ATMOSPHERES ON THE DUCTILITY OF T-111 TUBING AGED 2862 HOURS AT 1040° C (1900° F) IN VACUUM

[All samples prepared in air by hacksawing, filing, and dry sanding.]

Test atmosphere	Test results (a)
Air	Fractured
Argon	Ductile
Moist argon	Fractured
Dry air	Ductile
Nitrogen	Ductile
Oxygen	Ductile

[a]Ductility determined by tube flattening test at room temperature.

tened completely with no evidence of any cracking. Based on the test results, it appears that flattening the aged T-111 ring specimens in an atmosphere containing moisture can result in brittle fracture. Upon flattening, clean T-111 surfaces can be generated, possibly as microcracks at the sharp edges of the ring specimens. The moisture in the test atmosphere could react with these clean surfaces and could cause hydrogen embrittlement.

Hydrogen pickup during sample preparation. - In an attempt to support the hypothesis that the observed embrittlement was caused by hydrogen pickup after aging, two sets of ring specimens were cut to varying widths using a water-cooled cutoff saw. The purposes of this experiment were to determine if hydrogen is picked up during wet cutting and to determine if a hydrogen gradient exists between the center and the cut edges of the T-111 rings. The width of the rings varied from about 0.05 to about 0.5 centimeter (0.02 to 0.2 in.). Varying the ring width resulted in different cut-surface-area to volume ratios. Thus, if hydrogen was picked up and concentrated near the cut surfaces, the apparent bulk hydrogen content should increase with decreasing sample width. One set of rings was cut from the T-111 capsule that was aged 1128 hours at 1040° C (1900° F) in vacuum. The other set of rings was cut from a sample of the as-received T-111 tubing.

The test results are shown in figure 5. The bulk hydrogen content increases with decreasing sample width for both sets of specimens. These results indicate that hydrogen is picked up during wet cutting and that the majority of the hydrogen was located near the

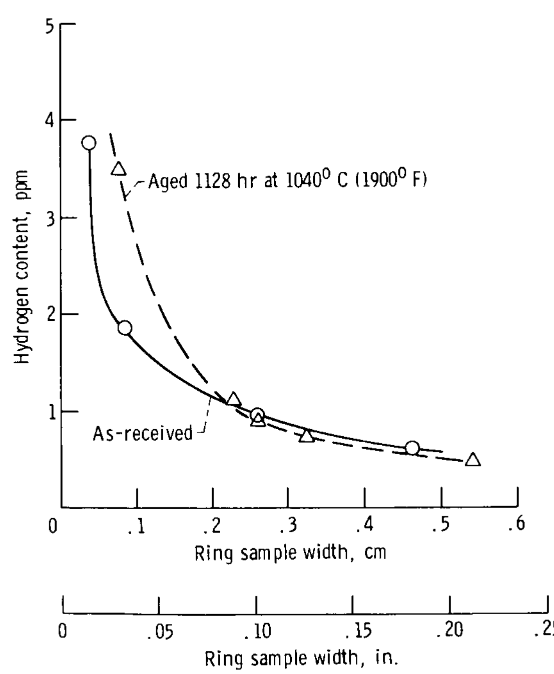


Figure 5. - Effect of ring width on hydrogen content of T-111 tubing specimens cut with water-cooled cutoff saw.

cut surfaces. Somewhat surprisingly, both the aged T-111 and the as-received T-111 picked up similar amounts of hydrogen. Therefore, aged T-111 must be considerably more sensitive to embrittlement than the as-received T-111. Although the amount of hydrogen picked up by the ring samples is relatively small, it could have a considerable effect on the ductility of the T-111 if it were concentrated at the grain boundaries.

Ductility of Sheet Specimens

Effect of aging. - The bend ductility of T-111 sheet specimens from each of the test capsules was determined from room temperature down to -196° C (-321° F). The sheet specimens were tested in the as-received condition and were not altered in any manner except for shearing to the 2.5 centimeter (1.0 in.) test length.

Most of the test specimens were quite ductile as indicated in table VIII. At room temperature all of the specimens could undergo a 180° 0t bend without cracking. At liq-

TABLE VIII. - EFFECT OF AGING ON THE BEND

TEST RESULTS OF T-111 SHEET

[All specimens bent at least 160°.]

Aging condition		Bend test temperature		Bend radius[a]	Test results
Hours at 1040° C (1900° F)	Contact with lithium	°C	°F		
0	No	24	75	0t	No cracking observed
		-196	-321	15	No cracking observed
0	Yes	24	75	0t	No cracking observed
		-196	-321	1t	No cracking observed
1128	No	24	75	0t	No cracking observed
		-196	-321	1t	No cracking observed
1128	Yes	24	75	0t	No cracking observed
		-196	-321	1t	
2862	No	24	75	0t	No cracking observed
		-196	-321	1t	No cracking observed
2862	Yes	24	75	0t	No cracking observed
		-106	-158	1t	No cracking observed
		-158	-252	15	Slight surface cracks
		-196	-321	1t	Edge and surface cracks

[a]Bend radius expressed in terms of the sheet thickness, t.

uid nitrogen temperature all of the specimens were ductile except for the samples from the sheet which was aged for 2862 hours in lithium. The behavior of even the specimens that cracked, however, was not the classic abrupt ductile to brittle transition with decreasing temperature. Instead, the amount of observed surface and edge cracking increased gradually with decreasing temperature. However, complete fracture was not observed.

Effect of wet sanding. - Samples of the as-received and aged T-111 sheet specimens were wet sanded to determine if the T-111 sheet was as sensitive as the T-111 tubing to hydrogen pickup. The sheet specimens were very lightly wet sanded on all surfaces with 400-grit silicon carbide paper. All specimens were wet sanded for about the same time (1 minute), and no measurable dimensional changes occurred as a result of sanding. The results of the 0t bend tests at room temperature are listed in table IX. Included in the table are the bend test results on the sheet prior to sanding.

TABLE IX. - EFFECT OF WET SANDING ON THE BEND TEST RESULTS OF T-111 SHEET

Condition		Test results[a]	
Time at 1040° C (1900° F), hr	Contact with lithium	Before sanding	After wet sanding
0	No	No cracking observed	No cracking observed
1128	No	↓	Deep edge and surface cracks
1128	Yes	↓	Deep edge cracks
2862	No	↓	Fractured after bending 90°

[a]All specimens (except fractured one) bent at least 180° over a 0t radius.

All of the specimens were ductile before sanding, whereas only the unaged T-111 was ductile after wet sanding. Apparently, the aged specimens picked up enough hydrogen during wet sanding to become embrittled.

Effect of bend test atmosphere. - The results of the bend tests on the aged T-111 sheet specimens did not show the sensitivity to the test atmosphere that was shown by the aged T-111 tubing samples. To investigate this difference in behavior, several additional tests were run on the T-111 sheet specimens. To duplicate the cut- and sanded-edge condition of the tubing specimens, the edges of 1040° C (1900° F) aged (1128 hr in lithium or 2862 hr in vacuum) T-111 sheet specimens were filed and dry sanded through 400-grit silicon carbide paper. Both specimens were ductile as determined by 180° 0t room temperature bend tests in air.

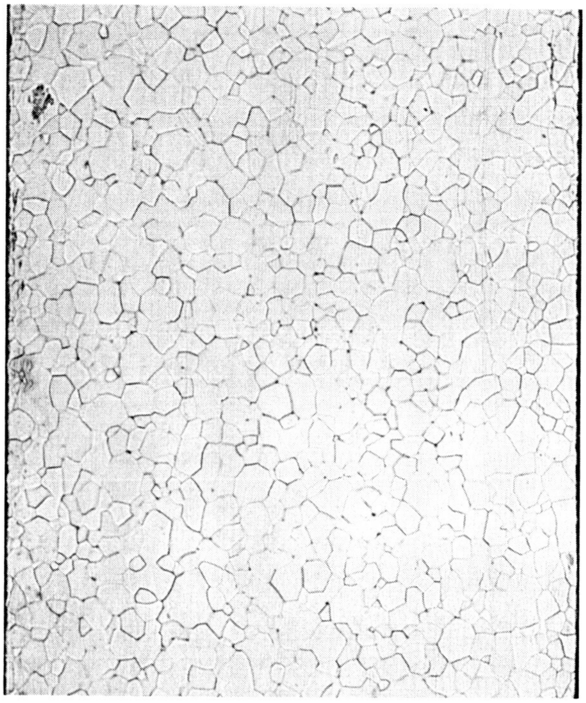
The sheet specimens that were aged at 1040° C (1900° F) for 2862 hours in vacuum were bend tested also in a moist argon atmosphere. One specimen was tested without any edge conditioning after aging, and the other specimen was sanded through 400-grit silicon carbide paper in moist argon prior to testing. Both specimens could sustain a 180° 0t bend at room temperature in the moist argon atmosphere without cracking. This indicates that the ductility of the aged T-111 sheet specimens is not as sensitive as the tubing samples to moisture in the test atmosphere.

Metallographic Examination

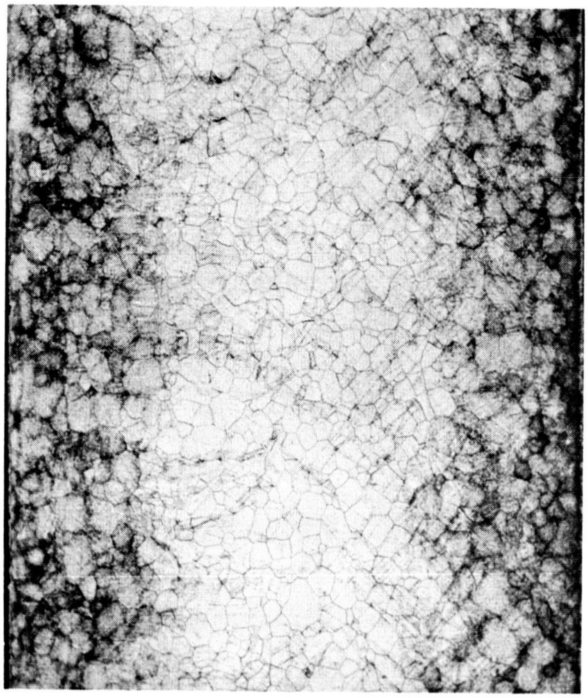
General metallography. - The microstructures of T-111 tubing samples in the as-received condition and after aging for 1128 and 2862 hours in vacuum at 1040° C (1900° F) are compared in figure 6. The as-received tubing is characterized by an equiaxed grain structure with no visible precipitates at this low magnification (X100). In contrast, 1040° C (1900° F) aging of the T-111 tubing produced dark bands of precipitate particles near the outside and inside surfaces (figs. 6(b) and (c)). No differences could be seen between the inside and outside surfaces of the tubing. The amount of precipitate particles observed after aging for 1128 and 2862 hours appears to be about the same for both conditions, which suggests that most of the precipitates were formed in less than about 1100 hours.

A comparison of the microstructures of the as-received material and 1040° C (1900° F) aged material for both heats of T-111 sheet is shown in figure 7. In agreement with the microstructure of the tubing, the aging treatment resulted in the appearance of dark bands of precipitate particles near the surfaces of the T-111 sheet. Even though the sheet samples from heat 650087 were aged over twice as long as samples from heat 650085, the amount of precipitates in the samples from heat 650087 appears to be considerably less. Heat 650087 contained less hafnium and oxygen than heat 650085 (table II) which may account for the difference in quantity of precipitates observed in the two heats of T-111 sheet.

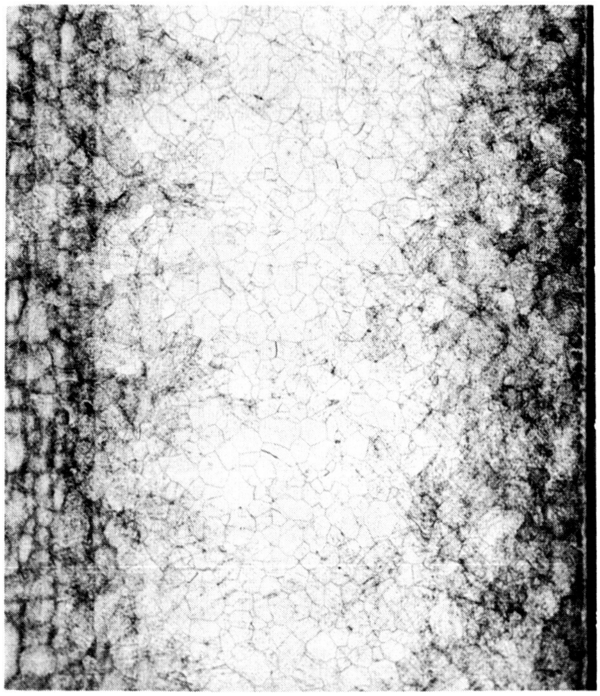
Particle observation. - Specimens were examined at a higher magnification using the light microscope and also by transmission and scanning electron microscopy to further characterize the precipitates that were observed in aged T-111. Figure 8(a) shows the microstructure of the as-received sheet from heat 650087. Only a few randomly distributed particles are observed. Effects of aging at temperatures of 980° and 1315° C (1800° and 2400° F) for times of 1000 hours or more are shown in figures 8(b) to (g). Aging at 980° C (1800° F), as shown in figures 8(b) and (c) resulted in formation of numerous precipitate particles located primarily at grain boundaries. In contrast aging at 1315° C



(a) As-received.

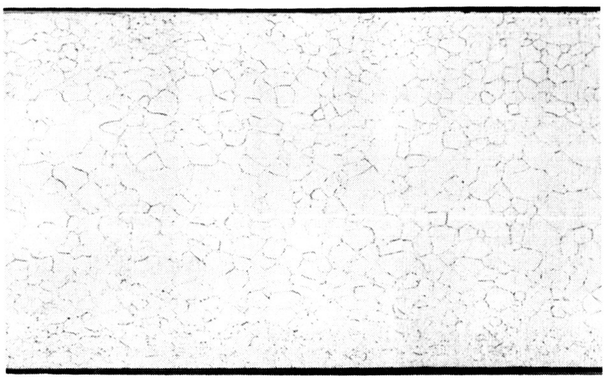


(b) Aged 1128 hours.

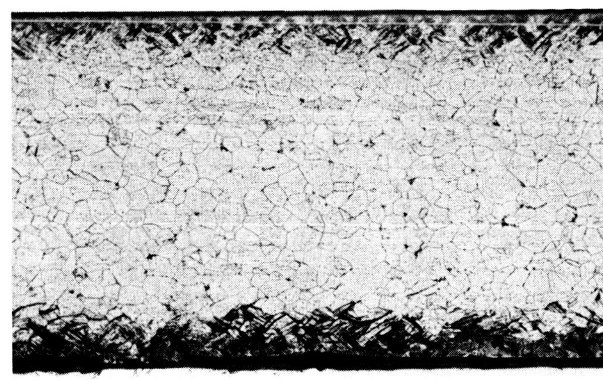


(c) Aged 2862 hours.

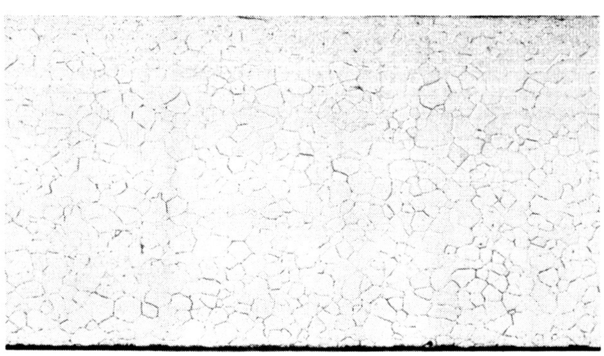
Figure 6. - Effect of aging at 1040° C (1900° F) in vacuum on microstructure of T-111 tubing. Longitudinal section; etchant, hydrofluoric acid, nitric acid, glycerine. X100.



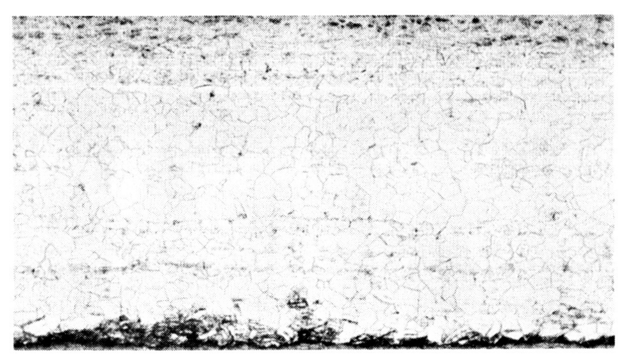
(a) As-received; heat 650085.



(b) Aged 1128 hours; heat 650085.

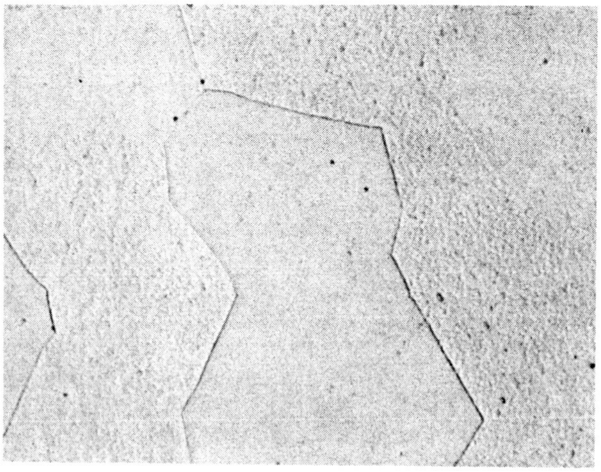


(c) As-received; heat 650087.

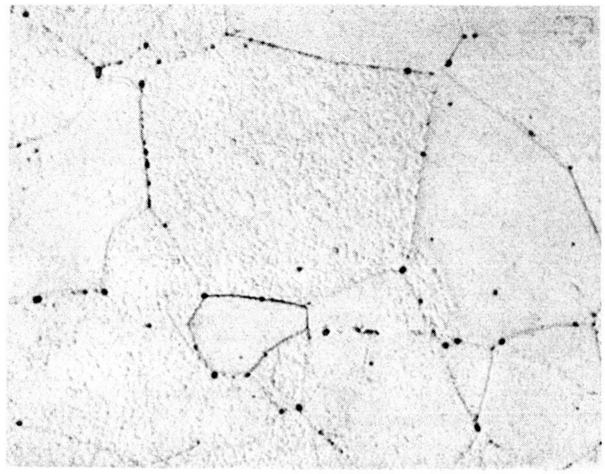


(d) Aged 2862 hours; heat 650087.

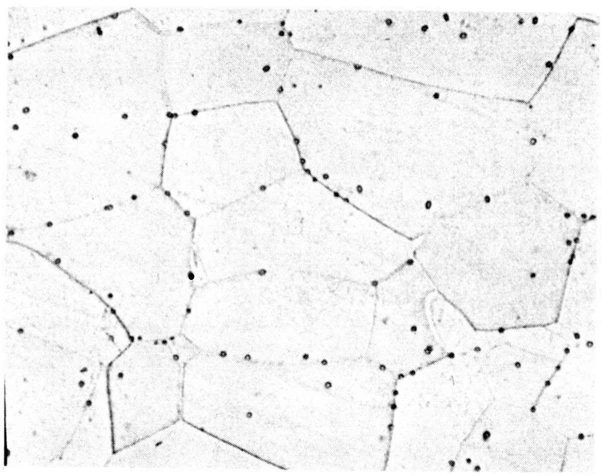
Figure 7. - Effect of aging at 1040° C (1900° F) in vacuum on microstructure of two heats of 0.051 centimeter (0.020 in.) T-111 sheet. Longitudinal section; etchant, hydrofluoric acid, nitric acid, and glycerine. X100.



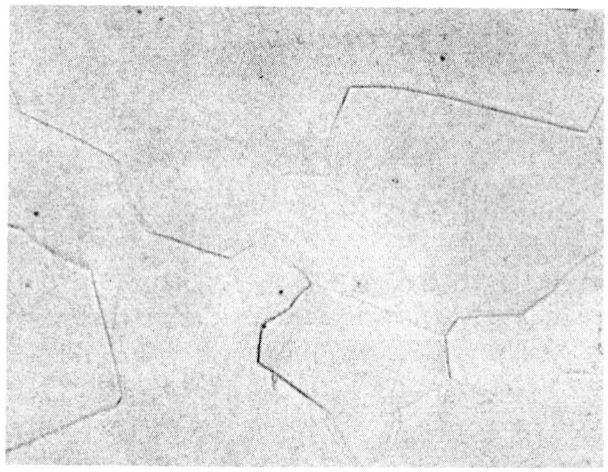
(a) As-received.



(b) Aged 1000 hours at 980° C (1800° F) in vacuum.

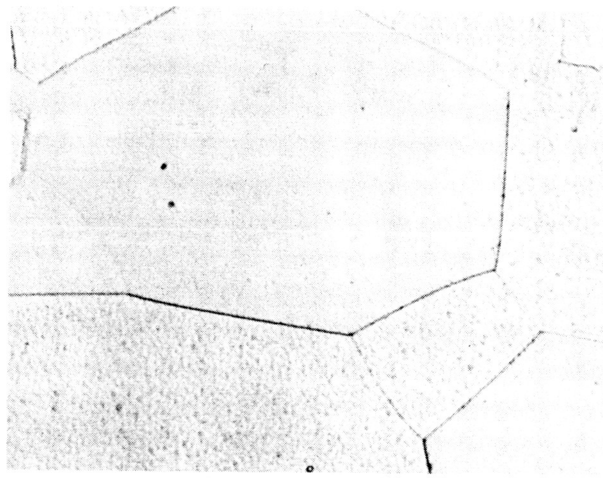


(c) Aged 1000 hours at 980° C (1800° F) in lithium.

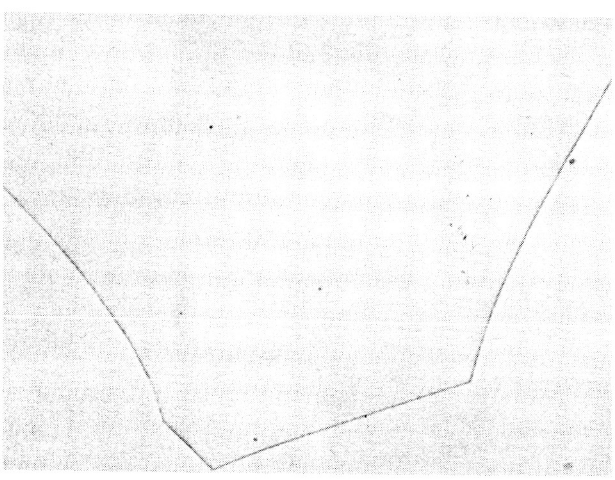


(d) Aged 1000 hours at 1315° C (2400° F) in vacuum.

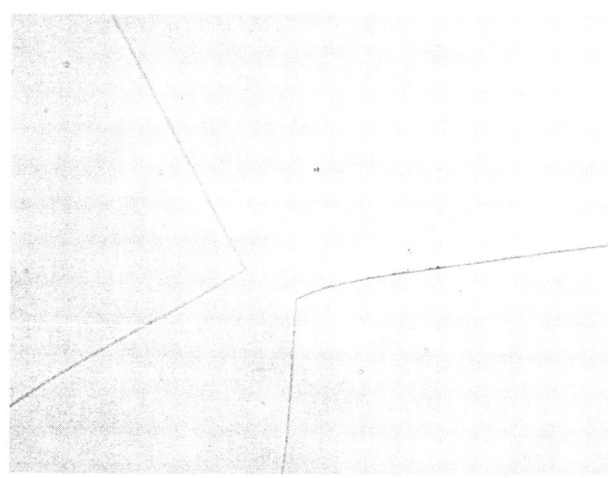
Figure 8. - Effect of aging temperature, time, and atmosphere on microstructure of T-111 sheet. Heat 650087; etchant, ammonium bifluoride, nitric acid, and water. X750.



(e) Aged 1000 hours at 1315° C (2400° F) in lithium.



(f) Aged 5000 hours at 1315° C (2400° F) in vacuum.



(g) Aged 5000 hours at 1315° C (2400° F), in lithium.

Figure 8. - Concluded.

(2400° F) resulted in a microstructure similar to the as-received sheet (figs. 8(d) and (e)) with these microstructures being characterized by equiaxed grains containing only a few precipitate particles. Considerable grain growth occurred upon extended aging for times up to 5000 hours at 1315° C (2400° F) (figs. 8(f) and (g)). This is a further indication that precipitate particles that would retard grain boundary movement were not present in significant quantity in the T-111 aged at 1315° C (2400° F). It should also be noted in figure 8 that the aging environment, vacuum or lithium, appears to have a minimal role in affecting the microstructure of T-111 aged over the temperature range 980° to 1315° C (1800° to 2400° F).

Transmission electron microscopy also was used to examine sheet T-111. Figure 9(a) shows that the as-received material had numerous dislocations but was free of precipitates. This foil was prepared from a disk cut from a bend test specimen, which accounts for the high dislocation density observed. Figures 9(b) and (c) shows that aging at 980° C (1800° F) for 1000 hours resulted in formation of rows of precipitate particles approximately 0.05 micrometers in diameter lying along grain boundaries. Aging these specimens for an additional 4000 hours at 1040° C (1900° F) resulted in fewer and larger particles being present at grain boundaries (figs. 9(d) and (e)). This suggests agglomeration of particles with extended aging time. The average particle size was approximately 0.3 micrometer after the extended aging treatment. Transmission electron microscopy observations on T-111 sheet aged for 1000 hours at 1315° C (2400° F) failed to reveal any precipitate particles (figs. 9(f) and (g)), confirming the light microscope observations that precipitation of grain boundary particles did not occur in T-111 during aging for several thousand hours at 1315° C (2400° F).

Scanning electron microscopy was used to observe particle morphology and distribution in T-111 tubing. Specimens were cut from tubing that had operated for 10 000 hours with potassium flowing through the tube while the outside diameter was exposed to a vacuum environment (ref. 4). Specimens cut from portions of the tubing that operated at 1040° C (1900° F) and 1200° C (2200° F) were prepared metallographically. Figure 10(a) shows the microstructure of the 1040° C (1900° F) portion of the tubing where numerous particles approximately 0.5 to 0.8 micrometer in diameter are noted to line the grain boundaries. A comparison of this particle size with particle sizes observed in aged T-111 sheet (figs. 9(b) and (d)) suggests that particle size continues to increase with increase in aging time at 1040° C (1900° F). A portion of the tubing that operated at 1200° C (2200° F) is shown in figure 10(b) where particles are observed at grain boundary triple points only, suggesting that 1200° C (2200° F) is near the maximum temperature where precipitation during aging occurs in T-111.

Particle identification. - An electron microprobe was used to identify the grain boundary precipitates observed in T-111 tubing and sheet aged at 1040° C (1900° F). Because of the small particle size compared with the electron microprobe beam diameter, it was

(a) As-received.

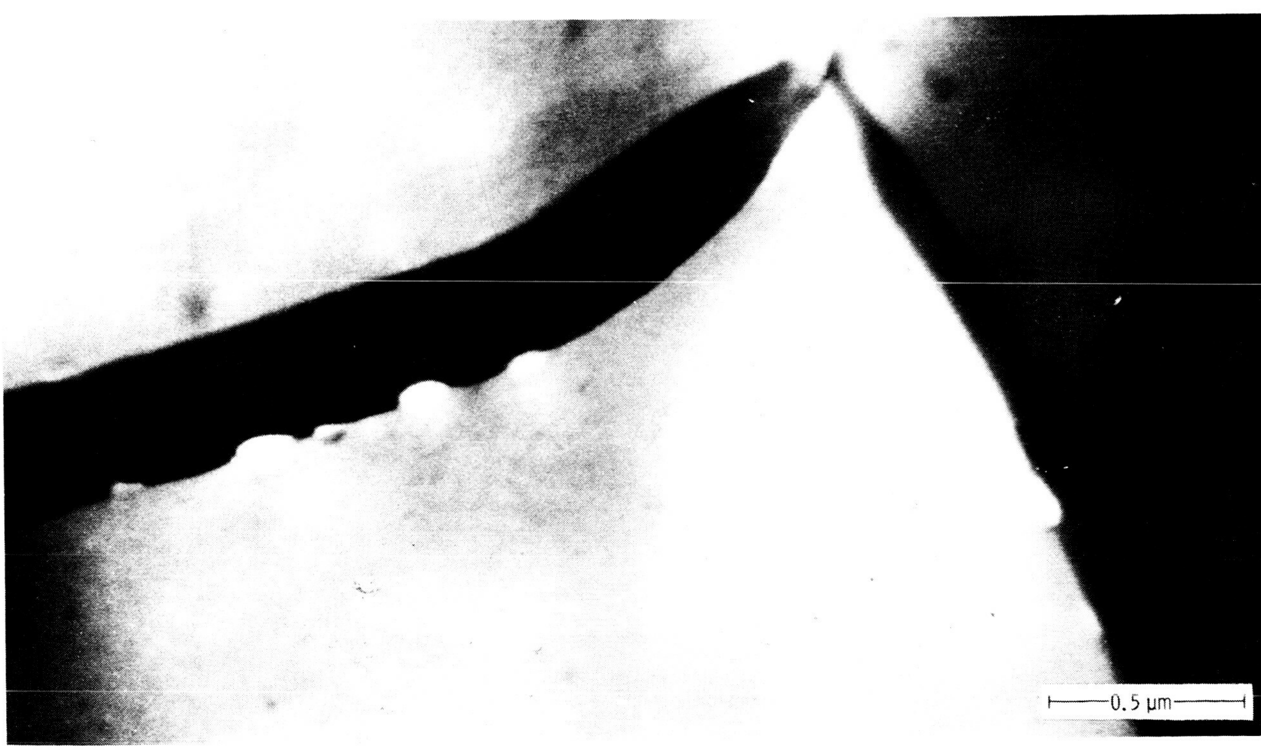
Figure 9. - Transmission electron micrographs of as-received and aged T-111 sheet.

(b) Aged 1000 hours at 980° C (1800° F) in vacuum.

(c) Aged 1000 hours at 980° C (1800° F) in lithium.

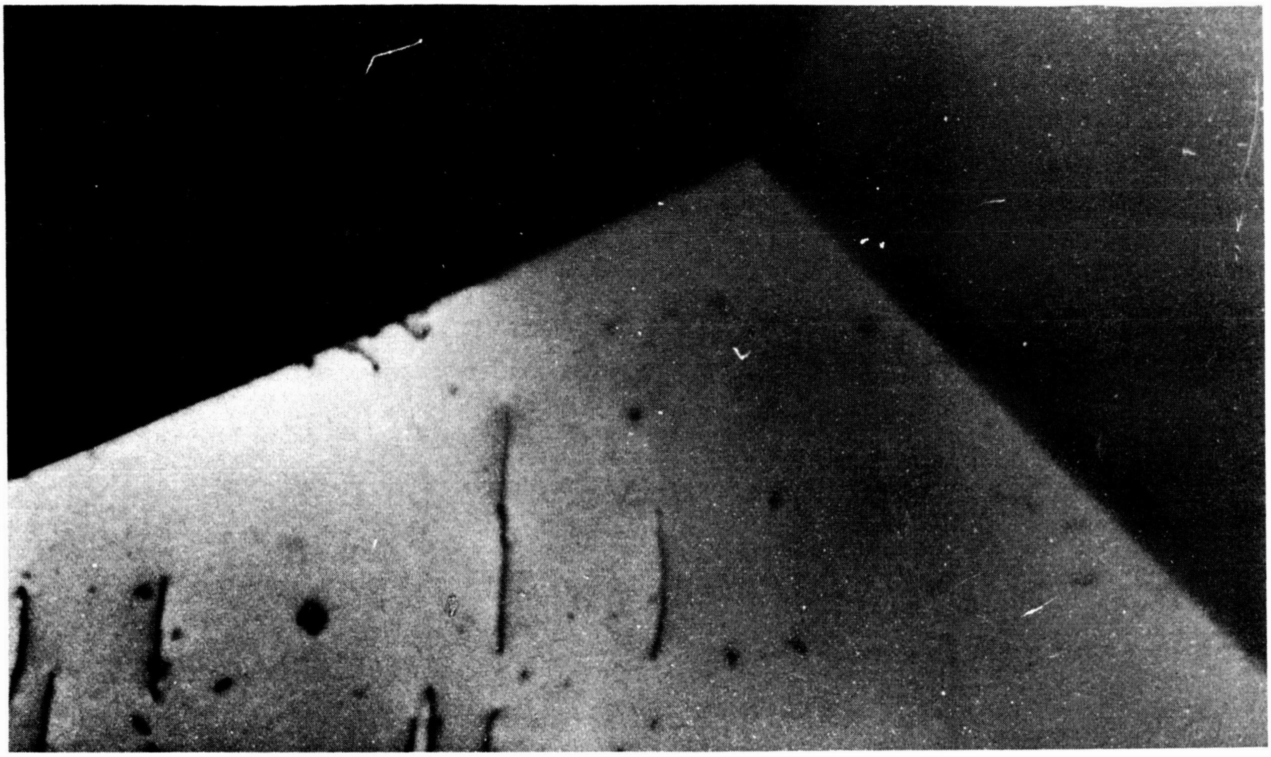
Figure 9. - Continued.

(d) Aged 1000 hours at 980° C (1800° F) and 4000 hours at 1040° C (1900° F) in vacuum.

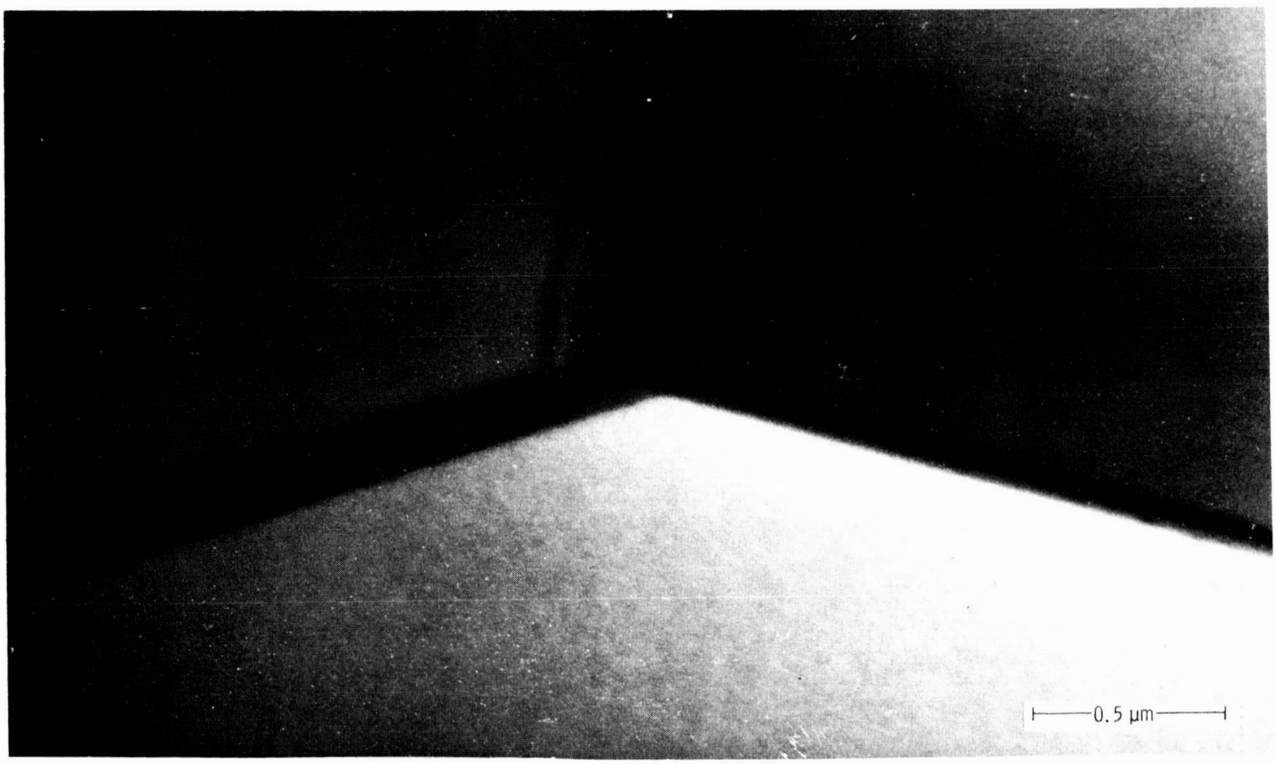


(e) Aged 1000 hours at 980° C (1800° F) and 4000 hours at 1040° C (1900° F) in lithium.

Figure 9. - Continued.

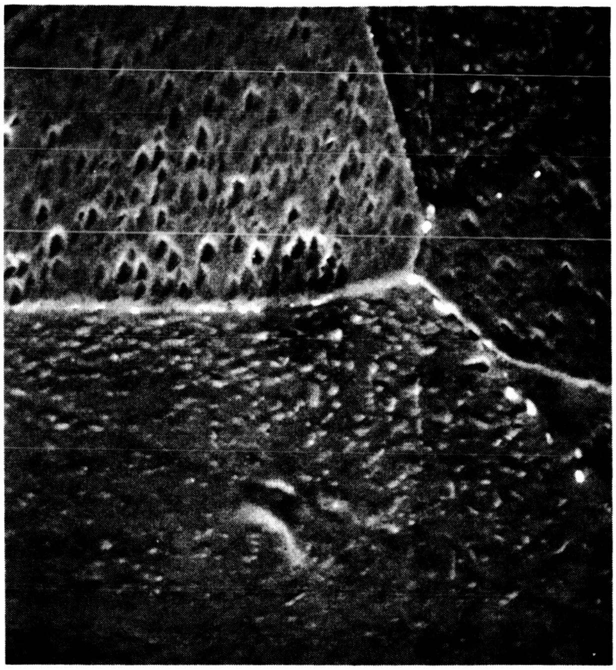


(f) Aged 1000 hours at 1315° C (2400° F) in vacuum.



(g) Aged 1000 hours at 1315° C (2400° F) in lithium.

Figure 9. - Concluded.



(a) 10 000 hours at 1040° C (1900° F).

(b) 10 000 hours at 1200° C (2200° F).

Figure 10. - Scanning electron micrographs of aged T-111 tubing. Etchant, ammonium bifluoride, nitric acid, and water. X2500.

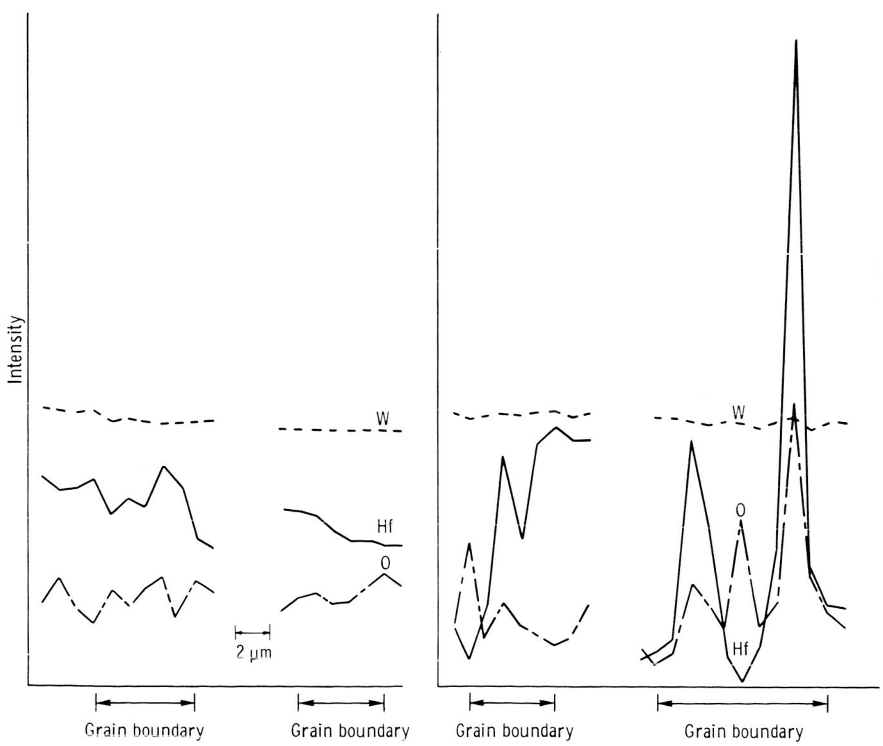


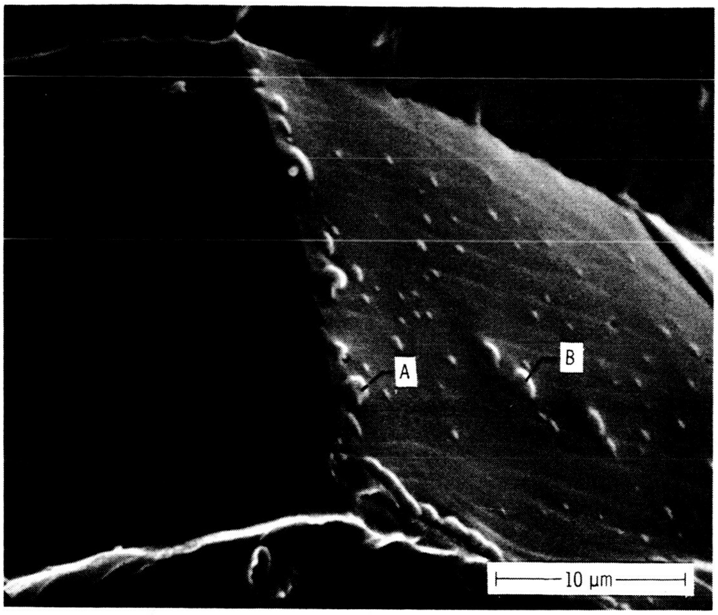
Figure 11. - Electron microprobe step-scan traverses of as-received and aged T-111 tubing.

not possible to analyze individual particles. But using a step-scan technique, qualitative information of the grain boundary constituents was obtained. Results of step-scan traverses along the length of grain boundaries in aged tubing are shown in figure 11. Several peaks of hafnium (Hf) and oxygen (O) were observed at grain boundaries in the aged specimens (10 000 hours at 1040° C (1900° F)) while only minor variations of Hf and O were observed in the as-received T-111 shown in figure 11(a). Tungsten peaks were not observed in either specimen as grain boundaries were traversed suggesting the absence of tungsten (W) segregation at grain boundaries. These results suggest that Hf and O segregate at grain boundaries in T-111 tubing during aging at 1040° C (1900° F). Segregation of Hf and O was not observed in the starting material. The uniformity of W in both specimens indicates that W_2Hf, which was postulated to be formed in welded and aged T-111 (ref. 3), is not the precipitate in aged T-111 tubing.

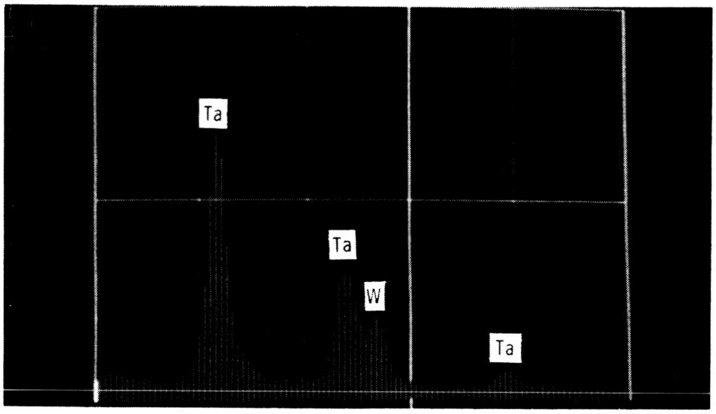
In order to confirm the identity of the grain boundary particles, characteristic X-ray analysis was performed in the scanning electron microscope using an energy dispersive spectrometer. Figure 12(a) shows the fracture surface of a sheet specimen aged 1000 hours at 980° C (1800° F) plus 4000 hours at 1040° C (1900° F). The specimen fractured at room temperature during bending. Particles are observed on the grain boundary surfaces and at grain boundary intersections. In addition, the brittle, intergranular fracture of aged T-111 is evident. Analysis of the grain boundary surface away from the particles is shown in figure 12(b). And figures 12(c) and (d) shows analyses of particles A and B in figure 12(a) located at a grain boundary intersection and on the grain boundary surface, respectively. The peaks for the elements detected are labeled in the figure. A comparison of the results from the two particles and from the grain boundary surface reveals that the particles exhibit Hf peaks, while these peaks are absent on the grain boundary surface. These results suggest that the grain boundary particles are Hf rich compared with the surrounding grain boundary surface.

A final analysis of the precipitate particles was performed on a T-111 sample aged for 10 000 hours at 1040° C (1900° F). This was done by dissolution in a bromine-methanol solution followed by X-ray diffraction of the residue. Results showed the particles to be HfO_2 plus a trace amount of Ta_2O_5.

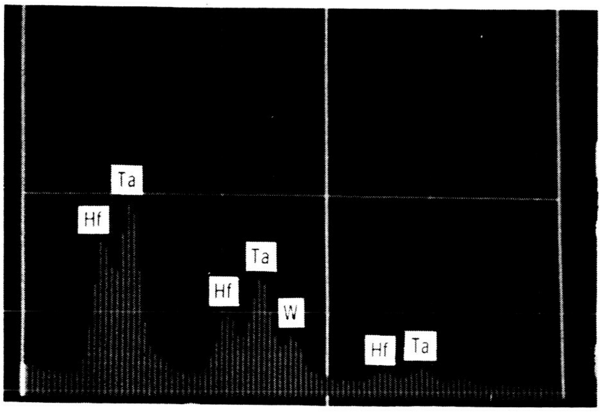
Based on this metallographic study of T-111 tubing and sheet, we conclude that aging T-111 for about 1000 hours at temperatures of about 980° to 1040° C (1800° to 1900° F) results in formation of HfO_2 particles located primarily at grain boundaries. Observations of metallographic cross sections as well as fractured surfaces show the presence of discrete grain boundary particles of HfO_2 in T-111 aged at 980° to 1040° C (1800° to 1900° F). In contrast, the starting material and specimens aged at 1200° to 1315° C (2200° to 2400° F) are essentially free of precipitate particles.

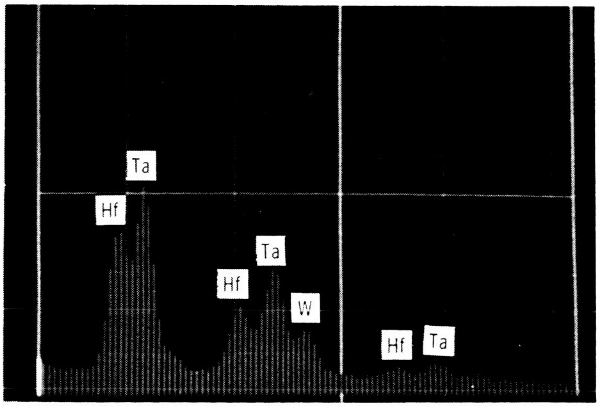


(a) Scanning electron micrograph.



(b) Grain boundary surface.





(c) Particle A.

(d) Particle B.

Figure 12. - Scanning electron microscope analysis of precipitate particles observed in 1040° C (1900° F) aged T-111 sheet.

DISCUSSION

All of the test results from this study indicate that both T-111 sheet and tubing that have been aged at temperatures near 1040° C (1900° F) are very sensitive to hydrogen embrittlement during post-aging handling operations. One source of hydrogen was shown to be contact of freshly-prepared T-111 surfaces with moisture during sample preparation after aging. The aged tubing specimens were more sensitive to test atmosphere than the sheet specimens. Aged tubing specimens tested in moist air fractured, whereas aged sheet specimens could undergo a 0t bend in moist air without fracturing. The reason for this difference in behavior is not known at this time, but it might be due to differences in hafnium content of the different lots of material. The tubing specimens contained considerably more hafnium than the sheet specimens (2.3 weight percent Hf in tubing against 1.8 or 2.0 weight percent Hf in the sheet).

The T-111 sheet, which was aged for 2862 hours at 1040° C (1900° F) in lithium, exhibited edge and surface cracking at -196° C (-321° F) when tested by bending. The specimen was tested in the aged condition without any cutting or surface preparation. But material aged at 1315° C (2400° F) for up to 5000 hours was ductile when tested under similar conditions. These observations suggest that reduced ductility of T-111 is directly related to aging at temperatures near 1040° C (1900° F) for several thousand hours.

In general, samples aged with and without contact to lithium were similar with respect to hydrogen sensitivity and microstructure. Thus, we conclude that the aging atmosphere used (either lithium or vacuum) does not significantly affect the sensitivity of T-111 to hydrogen embrittlement during post-aging operations. The aging temperature and possibly aging time appear to have much greater effects on post-aging embrittlement than does the aging environment.

A possible explanation for the loss in ductility of T-111 after aging at about 1040° C (1900° F) is associated with the HfO_2 precipitates at grain boundaries. Precipitates were observed in T-111 sheet and tubing aged at this temperature, while specimens that were aged at higher temperatures (near 1315° C (2400° F)) were ductile and were free of precipitate particles. Post-aging surface preparation (wet cutting or wet sanding) or testing in air probably aggravates the problem by introducing hydrogen into the T-111. This hydrogen apparently is concentrated at grain boundaries as indicated by the fact that grain boundary fractures were observed in hydrogen-embrittled T-111. Since it was shown that aged T-111 picks up no more hydrogen than the as-received T-111 under similar wet-cutting conditions, embrittlement by hydrogen in the material aged at 1040° C (1900° F) may be due to some free Hf or Hf-rich oxide particles at the grain boundaries. The presence of Hf or Hf-rich particles could concentrate hydrogen at grain boundaries which could lead to grain boundary failure. Alternately, hydrogen embrittlement may be an additive effect to the observed increase in ductile-brittle transition temperature resulting from the presence of HfO_2 particles.

Although we attribute the brittle behavior of the aged T-111 on hydrogen embrittlement, direct analytical results were inconclusive in identifying hydrogen as the cause of the embrittlement. These results were not unexpected since a small amount of hydrogen at the grain boundaries would not be detectable using conventional analytical techniques. The hydrogen embrittlement hypothesis, however, was supported by the other test results. For example, aged T-111 samples exposed to moisture during preparation or testing were brittle while aged samples not exposed to moisture were ductile. The fact that the ductility of the brittle T-111 samples could be restored by a relatively low-temperature vacuum anneal also supports the hydrogen embrittlement hypothesis.

Although the exact role played by hydrogen in embrittling T-111 is not known, embrittlement of unalloyed tantalum by hydrogen is well documented and has been reviewed recently by Chandler and Walter (ref. 7). Tantalum falls into a classification termed "exothermic occulders of hydrogen" along with other metals such as titanium, hafnium, and columbium. Exothermic occulders are characterized by a positive heat of absorption and by formation of hydrides at low temperatures. In contrast, endothermic occulders of hydrogen such as tungsten, iron, and copper have a negative heat of absorption and do not form hydrides. Hydrogen embrittlement of tantalum alloys can thus be expected to arise from three possible mechanisms: (1) hydrogen in solution, (2) hydride formation, and (3) interaction between hydrogen and an alloying element. Examples of the first two mechanisms have been reported in the literature for unalloyed tantalum and for the T-111 alloy. Small amounts of hydrogen in solution have been shown to produce embrittlement in unalloyed tantalum. For example, Nunes et al., (ref. 8) have reported a ductile-to-brittle transition temperature of approximately -130° C (-202° F) for tantalum containing only 7 ppm hydrogen. The T-111 alloy was shown by Stephens and Garlick (ref. 9) to be susceptible to the second mechanism, hydride embrittlement.

The present investigation cannot distinguish between the three possible mechanisms of hydrogen embrittlement. But, the brittle intergranular fractures and the presence of HfO_2 at the grain boundaries of aged and embrittled T-111 suggests that the third mechanism, interaction between hydrogen and an alloying element (Hf) in the T-111, may play a dominant role in the observed embrittlement. The other two mechanisms, however, cannot be completely excluded.

CONCLUDING REMARKS

The properties of T-111 (such as good fabricability, weldability, and adequate high temperature strength) still make it attractive for use in space power systems. This study has served to emphasize, however, the care that must be taken in handling, repairing, or modifying portions of T-111 systems that have operated near 1040° C (1900° F) for long periods of time. Our results indicate that the loss of room-temperature ductility, observed in T-111 that had been aged near 1040° C (1900° F), was caused by post-aging handling operations. Aging at about 1040° C (1900° F) appears to increase the sensitivity of T-111 to subsequent hydrogen embrittlement. During aging, the T-111 specimens were apparently ductile since, after aging, they could be flattened or bent at room temperature in a moisture free atmosphere without cracking. Thus, T-111 used for components of future space power systems can be expected to remain ductile under their normal operating conditions. It should be emphasized that the observed hydrogen embrittlement, caused by post-aging handling, occurs only at relatively low temperatures. At elevated temperatures, hydrogen embrittlement would not be a problem.

The presence of HfO_2 concentrated at the grain boundaries apparently does not hinder the application of T-111 in space power systems since loops have operated for times up to 10 000 hours (ref. 4) in the 1040° C (1900° F) temperature range without problems of embrittlement during operation or of liquid-metal corrosion. There is the possibility, however, that aging for times greater than 10 000 hours may result in the continued agglomeration and growth of HfO_2 particles, which could produce a further increase in the ductile-to-brittle transition to possibly near room temperature.

The embrittlement of unaged T-111 observed during processing and after cutting or machining operations (ref. 2) also may be due to hydrogen embrittlement. If during processing a microstructure sensitive to hydrogen embrittlement was produced in the T-111, exposure to moisture during subsequent operations could have caused embrittlement.

CONCLUSIONS

Samples of T-111 sheet and tubing were aged at 1040° C (1900° F) for up to about 3000 hours in either a vacuum or a lithium environment. Post-aging sample evaluation was concerned primarily with the effects of aging on ductility. Also, other T-111 samples similarly aged for up to 10 000 hours were extensively evaluated using various metallographic techniques. The following conclusions were drawn from this study:

1. Long-term aging of T-111 at about 1040° C (1900° F) increases the sensitivity of T-111 to hydrogen embrittlement during subsequent post-aging handling operations. Exposure of the aged T-111 to moisture during post-aging cutting or sanding operations can result in brittle, intergranular fractures under room temperature deformation. This potential problem can be avoided by preventing exposure of the aged T-111 to a source of moisture during post-aging processing or testing.

2. Aging T-111 near 1040° C (1900° F) results in extensive formation of HfO_2 located primarily at grain boundaries. These particles should not hinder the use of T-111 at elevated temperatures, but may account for the increased sensitivity of aged T-111 to subsequent hydrogen embrittlement.

3. The grain boundary hafnium oxide particles may be responsible for the loss of ductility seen in one aged T-111 sheet specimen at -196° C (-321° F). The majority of the aged T-111 sheet specimens, however, were ductile at -196° C (-321° F), when not exposed to a source of hydrogen.

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16. Abstract

The post-aging embrittlement of T-111 (tantalum - 8-percent tungsten - 2-percent hafnium) following exposure for up to about 10 000 hours at 1040° C (1900° F) in either vacuum or liquid lithium was investigated for sheet and tubing samples. This thermal aging was shown to greatly increase the sensitivity of T-111 to hydrogen embrittlement during subsequent room temperature specimen processing or testing. The hydrogen embrittlement problem can be avoided by preventing exposure to the T-111 to moisture during post-aging processing or testing. Aging at 1040° C (1900° F) also resulted in formation of HfO_2 particles at grain boundaries, which may contribute to the observed embrittlement.

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