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Work performed for
U.S. DEPARTMENT OF ENERGY
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PRELIMINARY STUDY OF NIOBIUM ALLOY CONTAMINATION BY TRANSPORT THROUGH HELIUM

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SUMMARY

Preliminary tests were conducted to determine if interstitial element transport through a circulating helium working fluid was a potential problem in Brayton and Stirling space power systems. Test specimens exposed to a thermal gradient for up to 3000 hr included Nb-1%Zr, a Sm-Co alloy (referred to as SmCo in this paper), Hiperco 50 steel, and alumina to simulate various engine components of the Brayton and Stirling systems. Results indicate that helium transport of interstitial contaminants can be minimized over a 7-yr life with a monometallic Nb-1%Zr design. Exposure with other materials indicated a potential for interstitial contaminant transport. Determination of contamination kinetics and the effects on structural integrity will require additional testing.

E-3401

INTRODUCTION

This report presents preliminary findings of an investigation requested by the SP-100 Office at the Lewis Research Center of the National Aeronautics and Space Administration (NASA) to determine if mass transport of gaseous contaminants is a potential problem for long-term (7 to 10 yr) operation of refractory metal Brayton and Stirling engines which use an inert gas as the working fluid.

In response to this request, a simplified test was designed to provide a quick order-of-magnitude estimate of the severity of the perceived problem. This report documents the preliminary findings of this effort.

PROCEDURE

Power System Condition Simulation

A simple thermal convection capsule design was chosen to simulate conditions with the Brayton and Stirling systems. Since the Brayton system has a unidirectional, closed-loop flow path, this choice would seem reasonable on a rough order-of-magnitude basis for determining whether significant transport of gaseous contaminants could occur to or from sensitive refractory alloys during extended operation.

Because the Stirling system operates with oscillating gas flow, the question of appropriateness of this capsule test was raised. Operators of Stirling systems were of the opinion that complete mixing of the gas occurs within a few minutes and certainly within an hour. On this basis it was concluded that this type of test would be a reasonable simulation for the Stirling system also.

Nb-1%Zr was chosen as the refractory alloy for this test because it was readily available and it also is a potential candidate for the working fluid containment material for high performance space versions of both these systems. Conceptually, all nonrefractory alloy materials which would be exposed to the working fluid could be coated or canned in the containment material refractory alloy. However, if any of these materials, for any reason, were exposed to the working fluid, they could be a source of contamination for the refractory alloy. Selection of nonrefractory alloy materials for inclusion in these tests was based on this worst-case scenario. It was decided to include representative materials for engine components such as magnets, alternator, and insulation. A Sm-Co alloy, referred to in this investigation as SmCo, was selected as the magnet material. Hiperco 50 was selected as the alternator material. Insulation was represented by high purity alumina. In addition, molybdenum was chosen as the test capsule material to prevent extraneous contamination from the test environment.

Specimen Preparation, Exposure, and Analysis

Five molybdenum tubes, and the specimens they were to contain, were prepared for testing in accordance with the flow chart outline in figure 1. Cleaning of all materials was accomplished using the procedures outlined in table I.

Cleaned specimens were then strung to molybdenum lids and to each other using molybdenum wire as shown in figures 2 and 3. Capsules containing only Nb-1%Zr specimens were included to provide a baseline for comparison and to determine the degree of redistribution of interstitial contaminants within the Nb-1%Zr after exposure to the temperature gradients used in these tests.

Gas tungsten arc welding (GTAW) was used to weld the lids to the tubes. The welding chamber containing the tubes, lids, and specimens was evacuated to 1×10^{-2} Pa, then backfilled with helium to a pressure of 1×10^{-5} Pa, for welding. Helium dew point, water vapor, and oxygen analysis were monitored before and after GTAW. Representative values are given in table II. A pressurized helium leak test was used on each welded tube before and after exposure to assure weld integrity during the experimental testing.

Figure 4 is a schematic representation of the tubes placed in the furnaces. Note that the tubes and furnaces are in vacuum chambers and that the tubes extend partially into the furnaces. Thermal gradients along the tubes were determined using Pt/Pt-13%Rh thermocouples along the tube wall.

Following removal from the tested tubes, selected Nb-1%Zr specimens had coupons removed by shearing (figs. 5 to 7) for Auger surface analyses. An as-cleaned coupon of untested Nb-1%Zr was included as a control specimen. All Auger analyses were performed by a single commercial laboratory, using argon-ion etching to determine the composition of the coupon as a function of depth below the surface.

The remaining portions of these specimens and the other Nb-1%Zr specimens shown in figures 5 to 7 were subjected to bend testing. Nb-1%Zr specimens not subjected to the capsule test were also tested as control samples. The bend test conditions were:

Temperature	Room temperature
Plunger radius, mm	1.6 (about 1T)
Span, mm	25
Crosshead speed, mm/min	25

Bulk chemical analyses for carbon, nitrogen, and oxygen were determined on each of the Nb-1%Zr bend-test specimens. In addition, analysis were also obtained on chips taken from each end of the long Nb-1%Zr specimen shown in figures 2 and 5. Analyses were also obtained on the Hiperco 50 and SmCo (figs. 3, 6, and 7). Alumina specimens were analyzed only for C. In all cases, untested starting materials were used as control specimens.

RESULTS

Bend Tests

All Nb-1%Zr specimens which were bend tested were totally free of cracking. No ductility loss was discerned for any of the specimens. A typical specimen after bending is shown in figure 8.

Bulk Chemical Analysis

Results of the analyses for interstitial elements in the as-cleaned and the tested materials are shown in table III. The average temperatures at the various locations on the molybdenum tubes are plotted against distance along the tube, along with the positions of the specimens within the tubes, in figures 5 to 7. The maximum changes in tested specimens as compared to carbon, nitrogen, and oxygen contents of as-cleaned specimens are shown in figure 9 for the 3000-hr tests.

Auger Surface Analysis

Auger analysis elemental concentrations were determined by multiplying the magnitude of the Auger signals by published sensitivity factors and dividing the result by the sum for all the measured elements. This technique is vulnerable to several sources of systematic error, but is useful as an approximation. When the coupons are all similar in composition, as is the case here, comparisons between coupons are valid even if the absolute concentrations are in error.

Three kinds of measurements were made on the depth profiles obtained from each coupon. First, the thickness of the contaminant layer on the surface was estimated by determining the sputter-etch time required for the Nb concentration to reach 90 percent of its maximum value. The etch time was approximately proportional to depth. This relationship was determined to be 15 nm/min. Next, the concentrations of C, O, and N were determined at a depth of 75 nm, well under the contaminant layer but still in the surface region of the metal. Finally, C, O, and N concentrations were measured in the region

200 to 300 nm below the surface. This depth is representative of the bulk of the metal. Plots of these concentrations as a function of depth are known as depth profiles. Figure 10 is a typical depth profile from one of the coupons. All but three coupons had surface layer thicknesses of 20 to 40 nm. Those three were about an order of magnitude thicker and were disregarded, as explained in the Discussion below. The remaining results are summarized in table IV and figure 11.

DISCUSSION AND CONCLUSIONS

Bend Tests

No change in bend ductility was observed after exposure of Nb-1%Zr to the test environment ranging from 430 to 1300 K and times up to 3000 hr.

It is believed that these capsule tests represent an overly severe simulation of Brayton and Stirling systems; that is, the ratio of the amounts of the available contaminants to the amount of sensitive refractory alloy in these tests far exceeded that which would exist in these engines. The purpose of these tests was not to determine the rate of contaminant transport, but to indicate whether a problem of structural integrity could occur in the Brayton and Stirling engines. From this aspect, the bend tests indicated no ductility loss.

Bulk Chemical Analysis

The short-term (750-hr) test with Nb-1%Zr only (tube A), resulted in a uniform C distribution and increasing N and O with increasing temperature (table III). All C and O levels were below the starting material, while N started at the as-cleaned level in the cold end and increased toward the hot end. The source of N and the location of lost C and O were not determined.

The second, long-term (3000-hr) test with Nb-1%Zr only (tube B), gave different results (table III and fig. 9). Carbon was at levels slightly under, or equal to, the as-cleaned value up to temperatures of about 1000 K. This lost C appeared to be transported to the hotter regions. Nitrogen followed a similar pattern, but levels higher than that of the as-cleaned material began as low as 1000 K. The behavior of O was the same as in the shorter-time test, but at somewhat higher levels at 1275 K and above with concentrations essentially the same as the as-cleaned material. The source of the extra carbon and nitrogen and the location of the lost oxygen were not determined.

Bulk analysis for C and O in the Nb-1%Zr specimens from the tests with other materials showed increases in all cases (table III and fig. 9). These specimens were located in the hot end of the tubes. Nitrogen results were ambiguous; the short time (tube C) and one long-time test (tube D) showed slight increases, while the other long-time test showed no increase above as-cleaned levels of N.

Bulk analysis of the SmCo showed a decrease in C level and no change in N in all cases, an increase in long-time O level, and no change in short-time O content (table III). The Hiperco 50 and alumina did not change significantly.

Therefore, the most likely source of O remained the SmCo, despite its higher post-test O levels. A possible explanation could come from post-test handling of SmCo prior to bulk analysis. No precautions were taken to prevent absorption of water vapor or reaction with air after removal from the molybdenum tubes. These specimens probably have very large surface areas for absorption and are known to be reactive in air in powder form. This could have lead to an erroneously high O analysis.

While the source of O and N is speculative, the C increases seen for Nb-1%Zr can be attributed directly to the SmCo. Although interstitial contaminants were shown to increase in the Nb-1%Zr specimens when they were tested with other materials, it is obvious that the levels were not high enough to cause problems in these specimens, since they were still fully ductile in bend tests. However, the question of reaction rate effects over a 7-yr life may need additional test time to resolve, since it represents a 20-fold increase in exposure time.

Auger Surface Analysis

Three depth profiles showed layer thicknesses an order of magnitude thicker than the others. The results were not errors in the depth profiles, since they were repeated on different spots on the same coupon surfaces and even on the other major surfaces of the coupons. These thick layers appeared to be unrelated to the test parameters and they occurred in both the absence and presence of other materials. There was no correlation with either test time or temperature in any one tube, and, in the cases in which test conditions were repeated, the radically different profile was not reproduced. Therefore, these effects were considered to be experimental artifacts that may be due to unusual contamination of specimens or tubes during preparation. Because of this possibility, it was felt that Auger data from those particular tubes were not reliable. These data were discarded in the treatment of the Auger results. It should also be noted that at concentrations below 1 percent, noise in the Auger spectrum contributed significantly to the signal. Thus, in table IV, levels reported as undetectable could represent concentrations as high as 1 percent.

Comparison of the as-cleaned specimens with the tested specimens (table IV) shows that the test exposure always reduced the thickness of the contaminant layer. The layer thicknesses are shown graphically in figure 11, where the effect is more apparent. From figure 11, it is also clear that longer test times always produced marginally thinner contaminant layers.

In all of the tested specimens, the O concentration at both measured depths is less than in the as-cleaned specimens. Generally, the C concentration is also lower. Although the concentration measurements are not very precise near 1 percent, there seems to be a tendency for the C and O concentrations to decrease with increasing temperature and time at temperature.

Generally, N is absent in all the specimens. The only exception to this seems to be the existence of marginally detectable amounts in the higher temperature, short-time tests. The N does not persist in the 3000-hr test specimens. Detected N could be due to residual N in the atmosphere in the tubes or as a contaminant in the specimens themselves. It must be kept in mind, however, that the level of N in table IV is at the limit of detection in the Auger spectrum and these observed variations may not be real.

The overall effect of the tests on the Nb-1%Zr specimens was a general tendency to clean the surfaces rather than contaminate them. The bulk concentrations of C, O, and N were at or below the limits of detection for the technique utilized. The test exposure condition probably permitted diffusion of any surface contamination into the bulk Nb-1%Zr, where the quantity of contamination could not be detected in the higher temperature specimens.

Implications for the Space System

The lack of firm evidence for transfer of interstitial contaminants either to or from Nb-1%Zr, in the absence of other materials, from either the cold or the hot end of tubes to the other by helium would indicate that such transfer is at most minimal and is not a legitimate concern as far as internal sources are concerned in a monometallic system.

The tests where other materials were included seem to indicate the potential of such transfer from nonrefractory alloy materials to Nb-1%Zr when ready sources are available. The severity of the potential problem has yet to be determined. A series of new tests is being devised in an attempt to estimate a worst-case contamination rate which could then be used to calculate an expected upper limit for a projected 7-yr operation.

However, even if the worst-case contamination rate had a significant effect on structural integrity, it would not rule out the use of these systems. Space engine designs for the Stirling or Brayton systems can include techniques to minimize the rate of contamination from such sources. For example, the magnets could be located outside the helium containment volume or, alternatively, they could be clad or canned in a material to contain the contaminants or minimize their rate of entry into the helium.

SUMMARY OF RESULTS

Preliminary tests were conducted to determine if interstitial element transport through a circulating helium working fluid was a potential problem in Brayton and Stirling space power systems. Molybdenum tubes, filled with helium and containing test specimens were exposed to a thermal gradient for up to 3000 hr. Test specimens included Nb-1%Zr, SmCo, Hiperco 50 steel, and alumina to simulate various engine components of the Brayton and Stirling systems. The temperature at the hot end was 1300 K while the temperature at the cold end varied from 430 to 810 K. Auger analysis, bulk chemical analysis, and bend testing of the Nb-1%Zr specimens were used to determine the presence and extent of interstitial element transport.

Results indicate that helium transport of interstitial contaminants can be minimized over a 7-yr life with a monometallic Nb-1%Zr design. Exposure with other materials indicated a potential for interstitial contaminant transport. Determination of contamination kinetics and the effects on structural integrity will require additional testing.

CONCLUSIONS

This report has presented the results of a preliminary investigation of helium transport of interstitial elements in similated Brayton and Stirling systems. The tests included Nb-1%Zr, SmCo, Hiperco 50 steel, and alumina specimens in molybdenum tubes with the hot ends at 1300 K and the cold ends at 430 to 810 K for test times up to 3000 hr. The following conclusions are offered on the basis of Auger surface analysis, bulk chemical analysis, and bend testing.

1. No transport problem was detected when Nb-1%Zr was tested alone, but a potential transport problem was detected when other materials were tested with Nb-1%Zr;

2. Bend testing showed no flaws, even for the specimens with the highest degree of contamination found: 88 ppm C and 200 ppm O;

3. Auger analyses were inconclusive;

4. Bulk chemical analyses yielded: (a) Insignificant helium transport of interstitial contaminants when Nb-1%Zr was tested alone, (b) insignificant N transport and detectable C and O transport when Nb-1%Zr was tested with other materials, (c) the major source of contaminants was the SmCo; and

5. Additional work is needed to define an upper limit on the contamination rate for Brayton and Stirling systems.

ACKNOWLEDGEMENTS

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TABLE I. - CLEANING PROCEDURES

Nb-1Zr Specimens
- 5 min ultrasonic cleaning in LOX-grade trichloroethane
- 15 min pickle in saturated NaOH at 323 K
- Sequential cleaning
  - Rinse in tap water
  - 5 min ultrasonic cleaning in distilled water
  - 5 min ultrasonic cleaning in ethanol
  - Air dry

SmCo and Hiperco 50 Specimens
- 5 min ultrasonic cleaning in LOX-grade trichloroethane
- 5 min ultrasonic cleaning in ethanol
- Air dry

Alumina Specimens
- 5 min ultrasonic cleaning in distilled water
- 5 min ultrasonic cleaning in ethanol
- Vacuum dry, 2 hr at 373 K

Molybdenum Parts
- 5 min ultrasonic cleaning in acetone
- Air dry

TABLE II. - TYPICAL HELIUM GAS ANALYSIS

	Supplier analysis	Tank sample	Chamber	
			Before welding	After welding
Concentration, ppm by volume				
Hydrocarbons	$^{a}1$	--	-----	-----
Neon	$^{a}23$	--	-----	-----
Water	$^{a}8$	--	<1	1.5
Hydrogen	$^{a}1$	--	-----	-----
Nitrogen	---	12	-----	-----
Oxygen	---	2	24	26
Dewpoint, K	---	--	194.4	198.9

[a] Maximum value.

TABLE III. - INTERSTITIAL ELEMENT ANALYSES OF TEST MATERIALS

(a) As-cleaned materials

Material	Carbon	Oxygen	Nitrogen
	Concentration, ppm by weight		
Nb-1%Zr	[a]18	[b]82	[b]38
SmCo	630	3200	280
Hiperco 50	58	84	37
Alumina	42	----	---

(b) Post-test analyses

Location	Temperature, K	Carbon	Oxygen	Nitrogen
		Concentration, ppm by weight		
Nb-1%Zr				
Tube A (750 hr)				
[c]1	775	11	61	39
2	775	11	62	40
3	815	12	68	43
4	870	10	61	41
5	980	9	63	44
6	1275	11	70	51
[d]1	1300	11	77	54
Tube B (3000 hr)				
[c]1	810	15	75	29
2	810	15	63	42
3	820	13	65	39
4	870	14	70	40
5	980	17	73	49
6	1275	21	82	67
[d]1	1300	31	86	63
Tube C (540-hr)				
1	1035	17	116	41
2	1035	15	97	41
3	1275	43	132	56
4	1275	35	128	45
Tube D (3000 hr)				
1	1035	33	98	45
2	1035	27	109	47
3	1275	48	128	64
4	1275	56	125	61
Tube E (3000 hr)				
1	770	20	91	30
4	770	18	79	35
2	1100	25	117	36
5	1100	33	180	28
3	1280	85	191	30
6	1280	88	200	40

[a] Average of 6 analyses.
[b] Average of 5 analyses.
[c] Top.
[d] Bottom.

TABLE III. - Concluded.

Location	Temperature, K	Carbon	Oxygen	Nitrogen
		Concentration, ppm by weight		
SmCo				
Tube C	775	320	3200	250
Tube D	820	280	3400	250
Tube E	460	430	3400	290
Hiperco 50				
Tube C	825	68	85	38
Tube D	850	64	101	38
Tube E	520	64	80	37
Alumina				
Tube C	875	31	----	---
Tube D	875	45	----	---
Tube E	580	42	----	---

[a] Average of 6 analyses.
[b] Average of 5 analyses.
[c] Top.
[d] Bottom.

TABLE IV. - DEPTH PROFILE RESULTS FROM Nb-1%Zr SAMPLES TAKEN FROM TUBES AND AS-CLEANED SPECIMENS

Tube	Time, hr	Temperature, K	Carbon	Oxygen	Nitrogen	Carbon	Oxygen	Nitrogen	Depth at 90 at % Nb, nm
			Concentration at 75 nm, at %			Concentration at 200 to 300 nm, at %			
(a)	----	----	1.7±1.2	4.2±1.7	(b)	1.7±0.24	1.7±0.24	(b)	35.2±13.8
A	750	770	3.5	0.6	(b)	1.3	(b)	(b)	30.1
A	750	930	.9	.5	0.5	(b)	.5	(b)	29.4
A	750	1300	1.3	(b)	.8	(b)	(b)	0.8	28.0
B	3000	810	2.6	↓	(b)	1.0	.5	(b)	25.5
B	↓	930	1.6	↓	↓	1.0	.5	↓	25.5
B	↓	1290	(b)	↓	↓	(b)	(b)	↓	<23.0
D	↓	975	.9	.9	↓	1.0	1.0	↓	<24.0
D	↓	1300	.9	(b)	↓	(b)	1.0	↓	<24.0

[a] As-Cleaned specimens.
[b] Undetectable level (not above background).

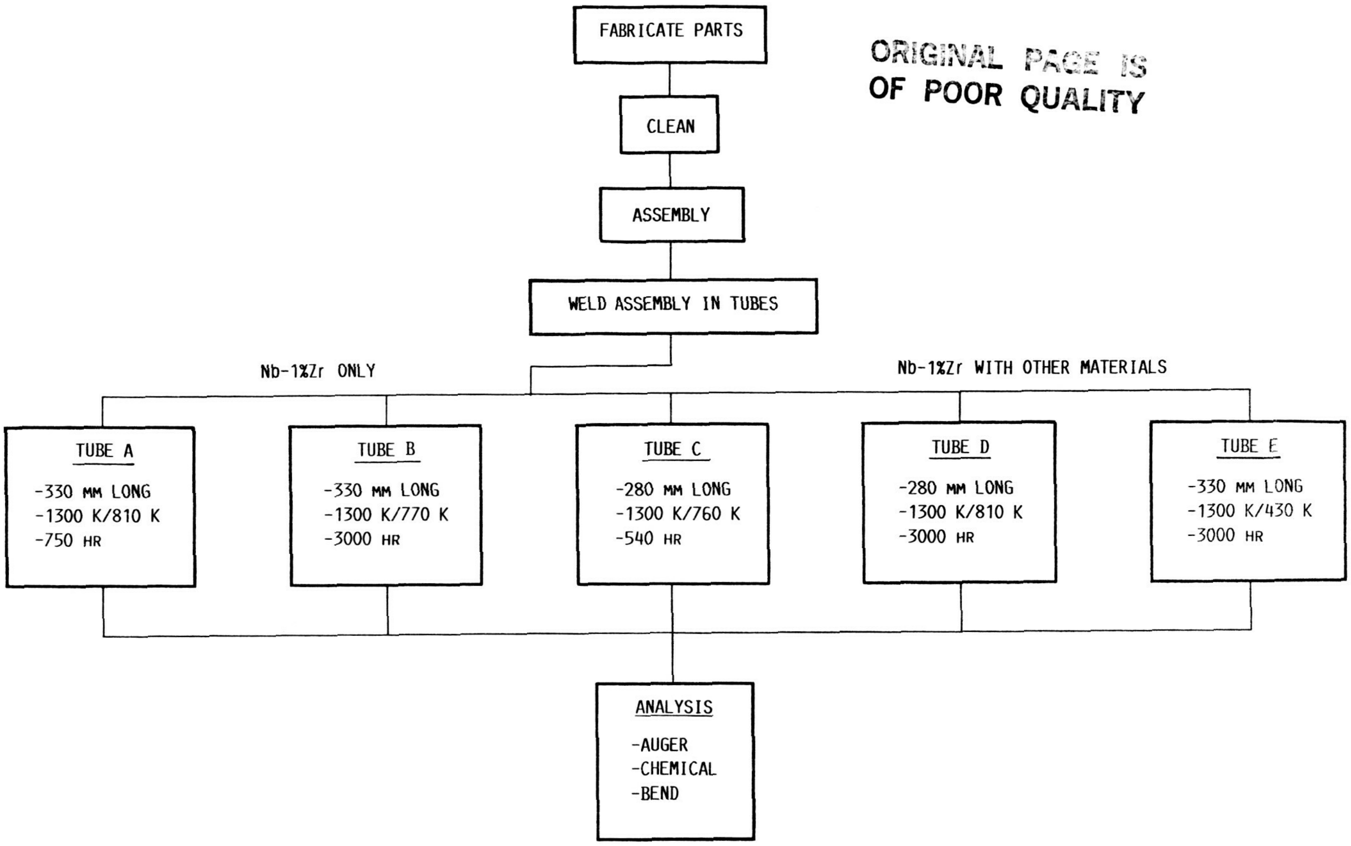

FIGURE 1.- FLOW CHART FOR HELIUM TRANSPORT STUDY.

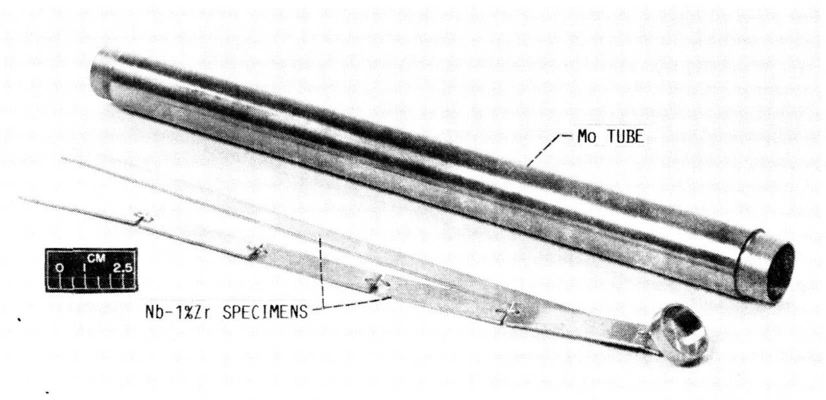

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FIGURE 2.- TYPICAL ASSEMBLY FOR TESTS WITH Nb-1%Zr SPECIMENS ONLY.

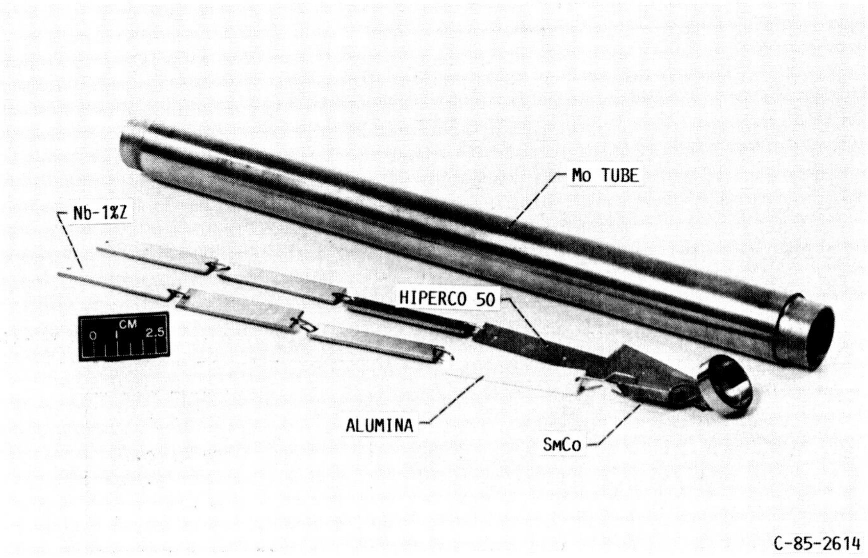

FIGURE 3.- TYPICAL ASSEMBLY FOR TESTS WITH Nb-1%Zr, HIPERCO 50, SMCo AND ALUMINA SPECIMENS.

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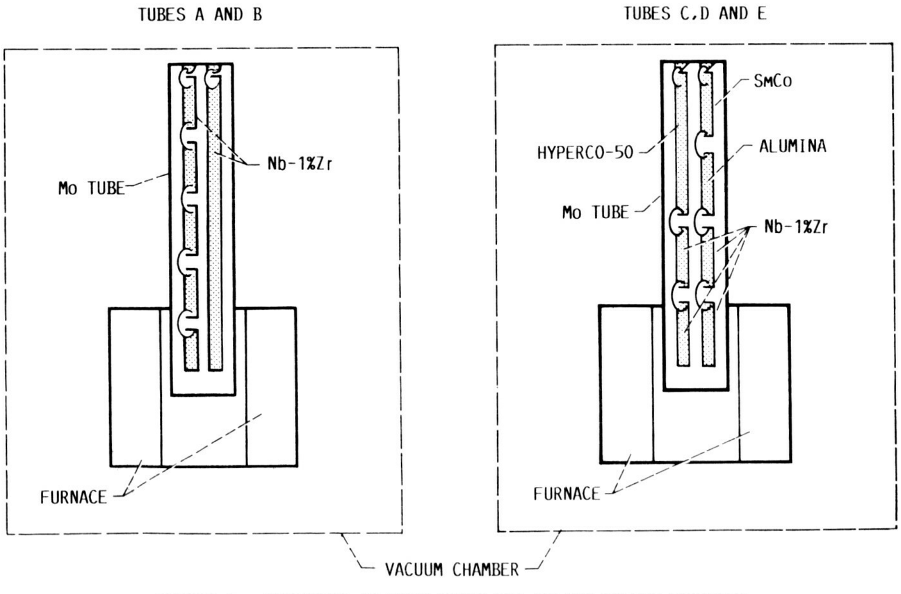

FIGURE 4.- SCHEMATIC OF TEST SETUP FOR HELIUM-FILLED CAPSULES.

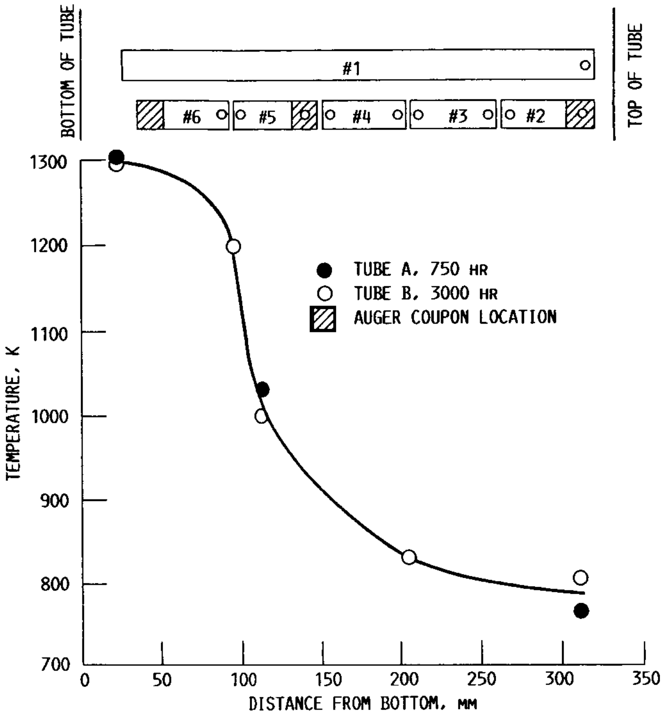

FIGURE 5.- THERMAL GRADIENT AND RELATIVE POSITIONS OF Nb-1%Zr SPECIMENS (TUBES A AND B).

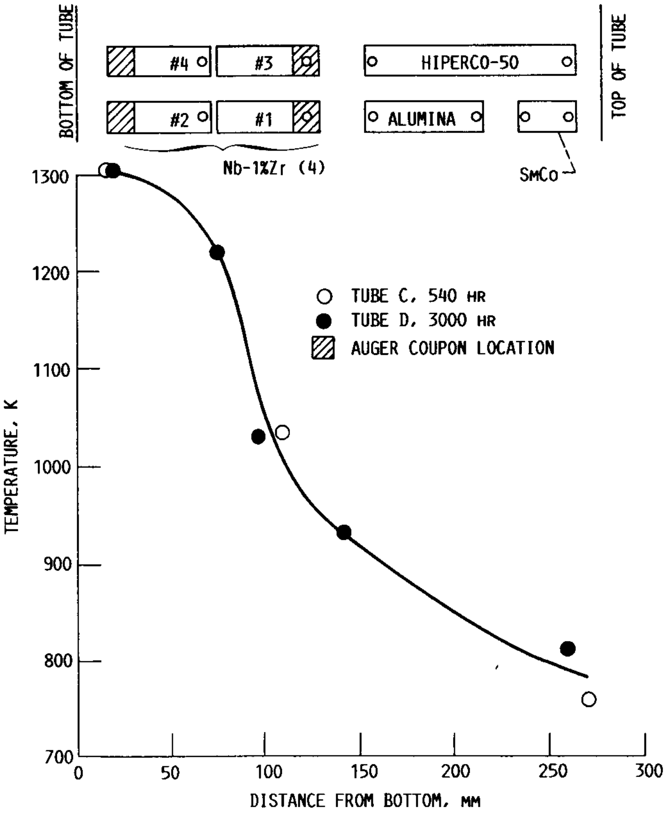

FIGURE 6.- THERMAL GRADIENT AND RELATIVE POSITIONS OF TEST SPECIMENS (TUBES C AND D).

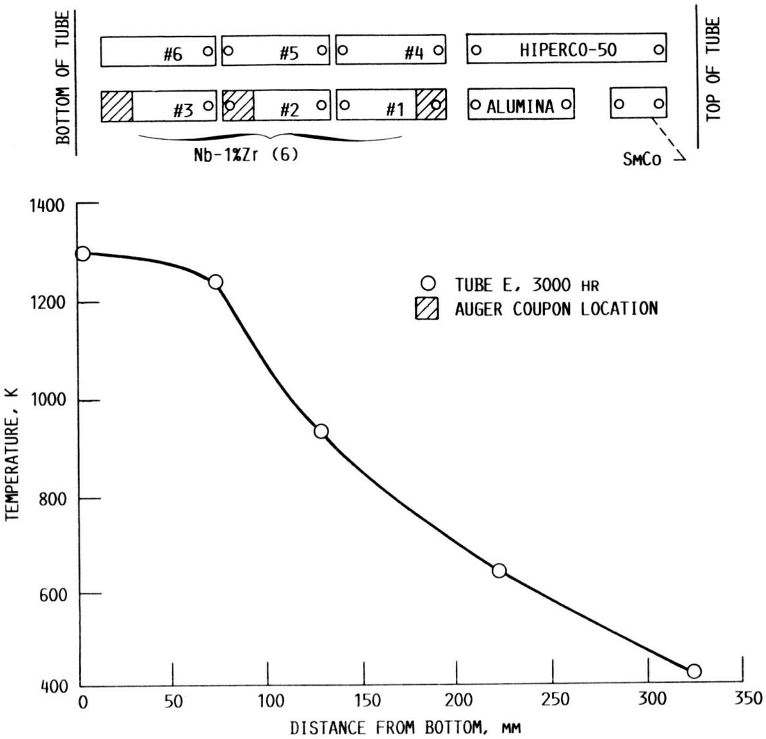

FIGURE 7.- THERMAL GRADIENT AND RELATIVE POSITIONS OF TEST SPECIMENS (TUBE E).

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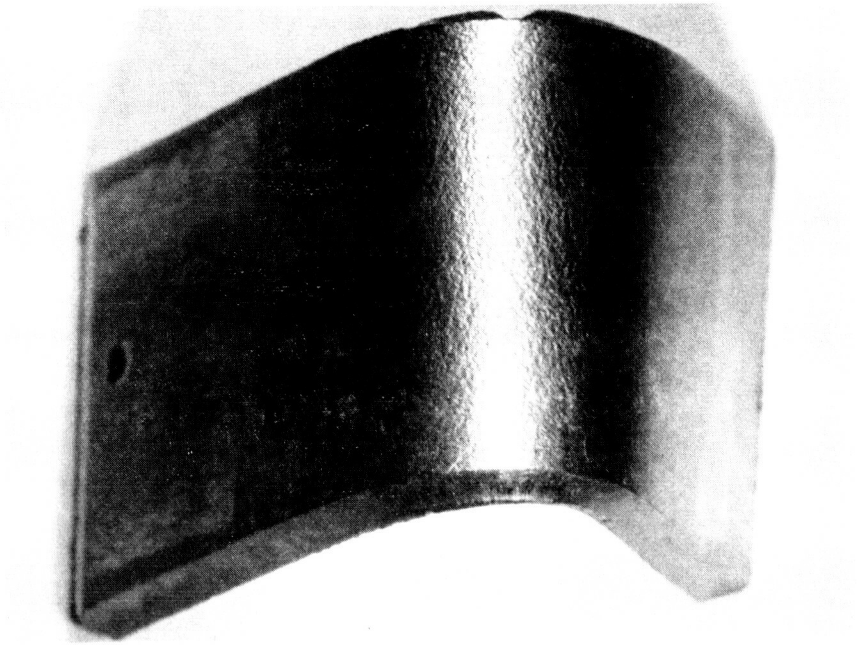
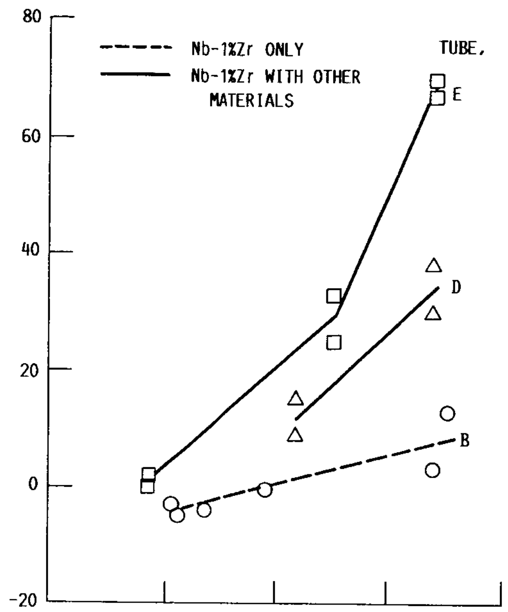
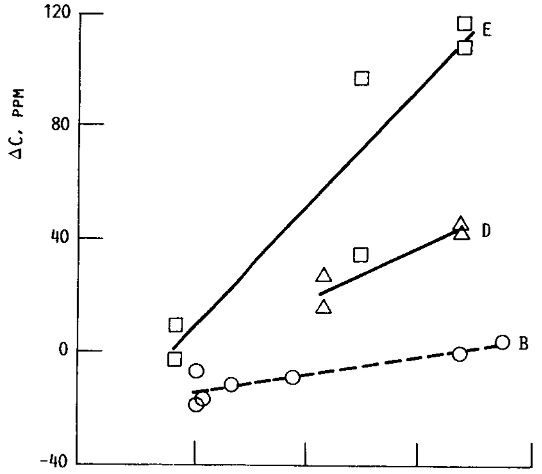


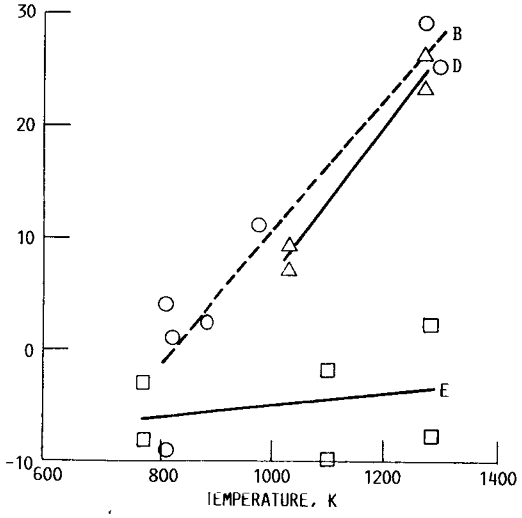
FIGURE 8.- TYPICAL Nb-1%Zr SPECIMEN AFTER BENDING.



(A) CARBON.



(B) OXYGEN.



(C) NITROGEN.

FIGURE 9.- RESULTS OF Nb-1%Zr BULK ANALYSES FOR 3000-HOUR TESTS.

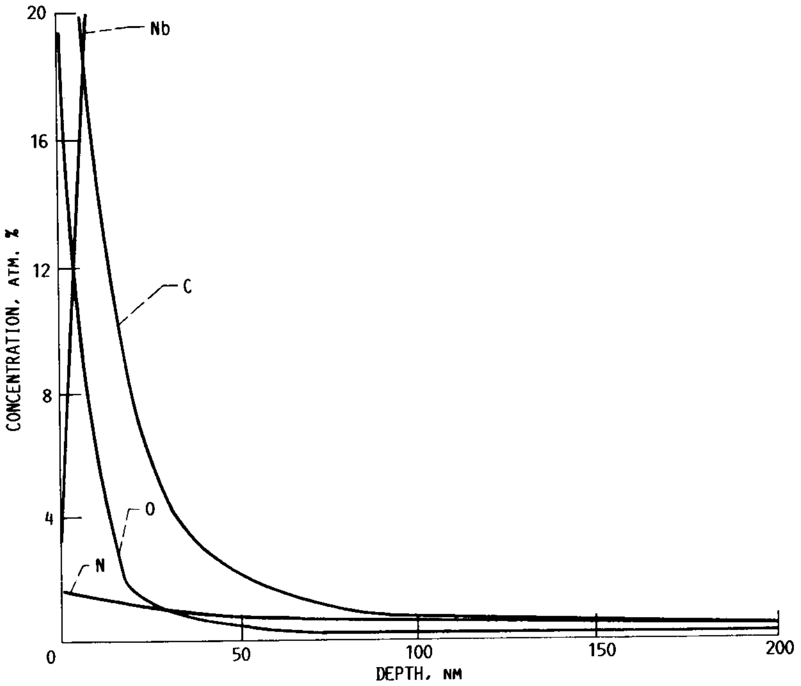


FIGURE 10.- TYPICAL AUGER DEPTH PROFILE.

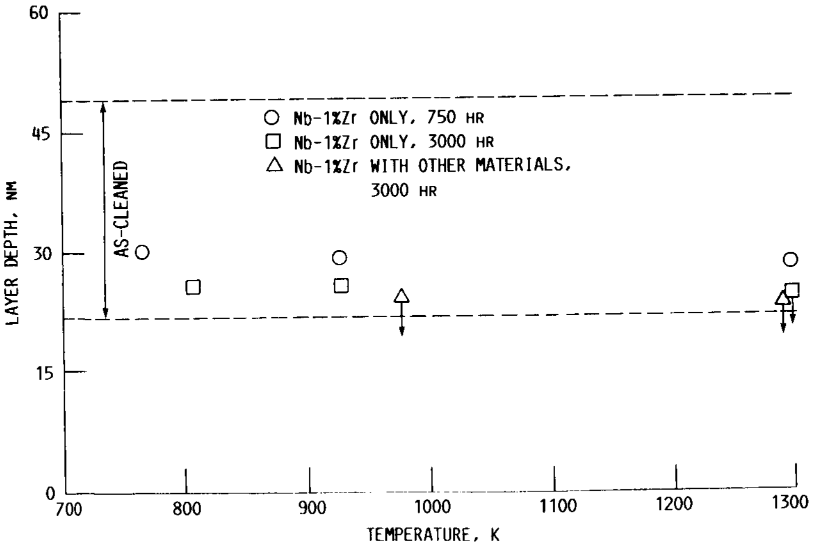


FIGURE 11.- EFFECTIVE LAYER DEPTHS ON Nb-1%Zr.

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16. Abstract
Preliminary tests were conducted to determine if interstitial element transport through a circulating helium working fluid was a potential problem in Brayton and Stirling space power systems. Test specimens exposed to a thermal gradient for up to 3000-hr included Nb-1%Zr, a Sm-Co alloy (referred to as SmCo in this paper), Hiperco 50 steel, and alumina to simulate various engine components of the Brayton and stirling systems. Results indicate that helium transport of interstitial contaminants can be minimized over a 7-yr life with a monometallic Nb-1%Zr design. Exposure with other materials indicated a potential for interstitial contaminant transport. Determination of contamination kinetics and the effects on structural integrity will require additional testing.

17. Key Words (Suggested by Author(s))
Refractory metals; Niobium alloys; Oxygen; Carbon; Nitrogen; Interstitial elements

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