WANL-PR(Q)-006

NASA CR54462

DEVELOPMENT OF DISPERSION STRENGTHENED TANTALUM BASE ALLOY

Fifth Quarterly Report

by

R. W. Buckman, Jr.

prepared for

National Aeronautics and Space Administration

Lewis Research Center

Space Power Systems Division

Under Contract (NAS 3-2542)

Astronuclear Laboratory

Westinghouse Electric Corporation

NOTICE

This report was prepared as an account of Government-sponsored work. Neither the United States nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

A) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately-owned rights; or

B) Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Astronuclear Laboratory

WANL-PR(Q)-006
CR-54462

DEVELOPMENT OF DISPERSION STRENGTHENED TANTALUM BASE ALLOY

BY

R. W. Buckman, Jr.

FIFTH QUARTERLY PROGRESS REPORT

Covering the Period

November 20, 1964 - February 19, 1965

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2542

Technical Management
Paul E. Moorhead
NASA-Lewis Research Center
Space Power Systems Division

Astronuclear Laboratory
Westinghouse Electric Corporation
Pittsburgh, Pa. 15236

ABSTRACT

30166

Development of dispersion strengthened tantalum base alloys for use in advanced space power systems continued with selection of the sheet and tubing alloy composition Ta-8W-1Re -0.7 Hf -0.025C for detailed evaluation. Scale-up of this composition to a four-inch diameter ingot was initiated. Selection of seven additional compositions in the Ta-Mo-Re-W-Hf-Zr-C-N system were made to further optimize the Ta-5.7 W-1.56 Re-0.7 Mo-0.25 Hf-0.13 Zr-0.015C-0.015N alloy. Response to heat treatment investigation continued and carbides and nitrides were shown to be stable in the tantalum alloy matrix at temperatures up to 1250°C and at pressures of 10^{-10} torr for 500 hours.

FOREWORD

This report was prepared by the Astronuclear Laboratory of the Westinghouse Electric Corporation under Contract NAS 3-2542. This work is administered under the direction of the Nuclear Power Technology Branch of the National Aeronautics and Space Administration with Mr. P. E. Moorhead acting as Technical Manager.

This work is being administered at the Astronuclear Laboratory by R. T. Begley, with R. W. Buckman, Jr. serving as principal investigator. This report covers the work performed during the period November 20, 1965 to February 19, 1965.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

I. INTRODUCTION

This, the fifth quarterly progress report on the NASA sponsored program, "Development of a Dispersion Strengthened Tantalum Base Alloy", describes the work accomplished during the period November 20, 1964 to February 20, 1965. This program is a continuation of the tantalum alloy investigation initiated under Contract NAS 3-2542.

The primary objective during this phase of work is the double vacuum arc melting of three compositions as 60 pound, 4 inch diameter ingots. Two compositions will be selected on the basis of a combination of weldability, creep resistance and fabricability. The third composition will be selected for optimum strength and fabricability for possible turbine applications.

During the Phase I investigation[1] several promising tantalum alloy compositions were developed which exhibited good resistance to creep deformation at 1315°C (2400°F) and still retained adequate fabricability and weldability. Improvement in creep resistance was achieved by a combination of solid solution and dispersed phase strengthening.

During this quarter, a weldable, carbide strengthened composition, NASV-20 (Ta-8W-1Re-0.7Hf-0.025C) was selected for scale-up and melting as a 4 inch diameter ingot. Seven compositions were selected for additional investigation of the strengthening resulting from the combination reactive metal carbide-nitride dispersion. They will be evaluated before composition selection and melting of the remaining two 4 inch diameter ingots. Weldability and creep strength under ultra-high vacuum conditions are the main evaluation criteria for sheet and/or tubing material, while creep strength and forgeability are the characteristics of interest for alloys for potential application in turbine components.

II. PROGRAM STATUS

A. OPTIMIZATION INVESTIGATION

Composition NAS-36 (Ta-5.7W-1.56Re-0.7Mo-0.25Hf-0.13Zr-0.015C-0.015N) developed during Phase I exhibited the highest creep strength at 1315°C (2400°F) of any of the tantalum alloys evaluated, and still retained good fabricability and weldability characteristics. Prior to melting this alloy as a 4 inch diameter ingot, the heats listed below were selected for investigation in order to optimize the composition.

NASV-12 (Ta-7.5W-1.5Re-0.5Hf-0.015C-0.015N)
NASV-13 (Ta-6.5W-2.5Re-0.3Hf-0.01C-0.01N)
NASV-14 (Ta-4W-1Mo-2Re-0.3Zr-0.015C-0.015N)
NASV-15 (Ta-9W-1.5Re-1Hf-0.06N)
NASV-16 (Ta-9.5W-0.5Re-0.25Zr-0.02C-0.01N)
NASV-17 (Ta-4W-3Re-0.75Hf-0.01C-0.02N)
NASV-18 (Ta-6W-1Mo-1Re-1Hf-0.01C-0.04N)

The above compositions will be double vacuum A. C. arc-melted into 2 inch diameter ingots weighing 7 pounds and then processed to 0.04 inch sheet via the schedule outlined in Figure 1. Evaluation will follow the schedule outlined in Figure 2.

Melting - First melt electrodes of compositions NASV-12, NASV-13, and NASV-14 were assembled using the sandwich technique which has been described previously[2]. The first melt was made into a 1-5/16 inch diameter mold. The first melt ingot was subsequently melted into a 2 inch diameter mold. All melting was accomplished using A. C. power. All the as-melted ingots had excellent sidewalls.

Primary Breakdown - A 3/4 inch section was removed from the bottom portion of each ingot and coated with an Al-12Si alloy and then upset forged at 1300°C (2372°F) on the Dynapak. All three compositions forged satisfactorily and the results are reported in Table 1. The balance of each ingot will be plasma sprayed with unalloyed molybdenum and extruded to sheet bar.

Secondary Working - The as-forged sheet bar was lathe conditioned, annealed for 1 hour at 1650°C (3000°F) and then processed to 0.04 inch sheet using the established practice. Excellent quality strip was produced from all three compositions.

Mechanical Property Evaluation - Creep testing under ultra-high vacuum conditions was initiated on heat NASV-14 and the results obtained during this period are reported in Table 2. A total of 4 creep tests will be run for each composition. The two temperatures at which each sheet and tubing composition will be evaluated are 1315°C (2400°F) and 1427°C (2600°F).

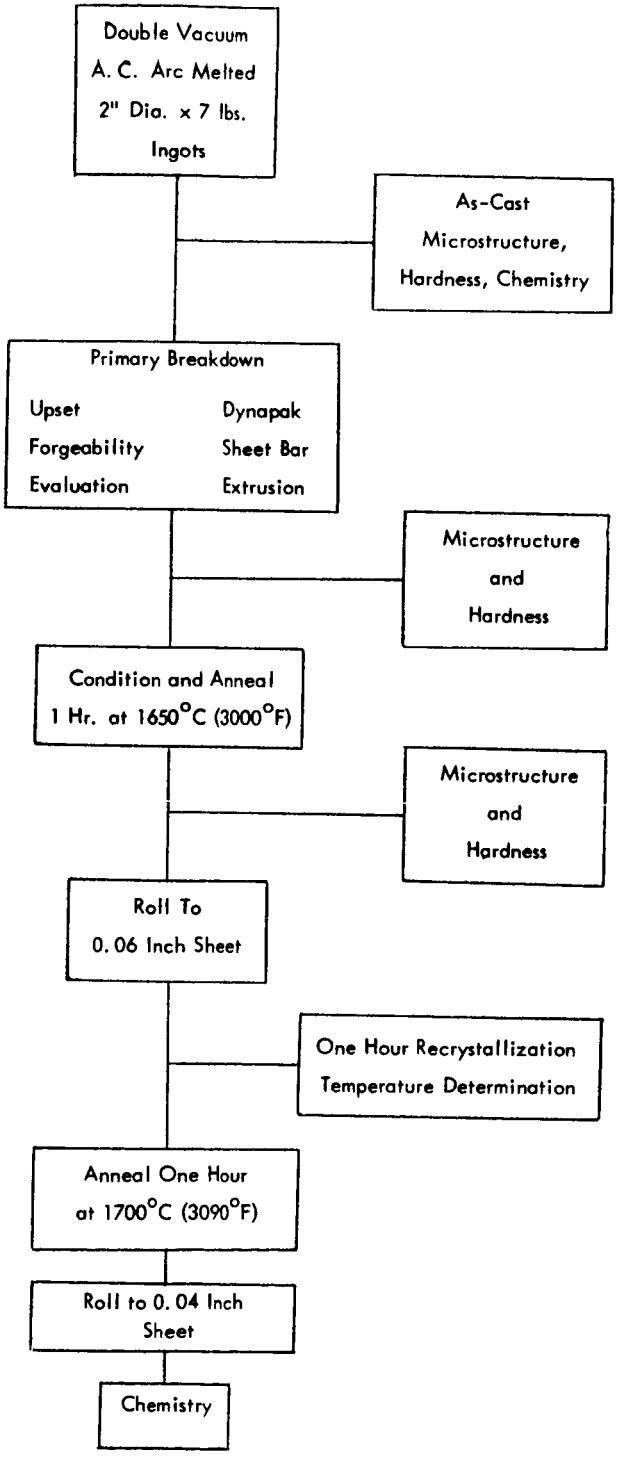


FIGURE 1 - Processing Schedule for Optimized Two Inch Diameter Ingot Compositions

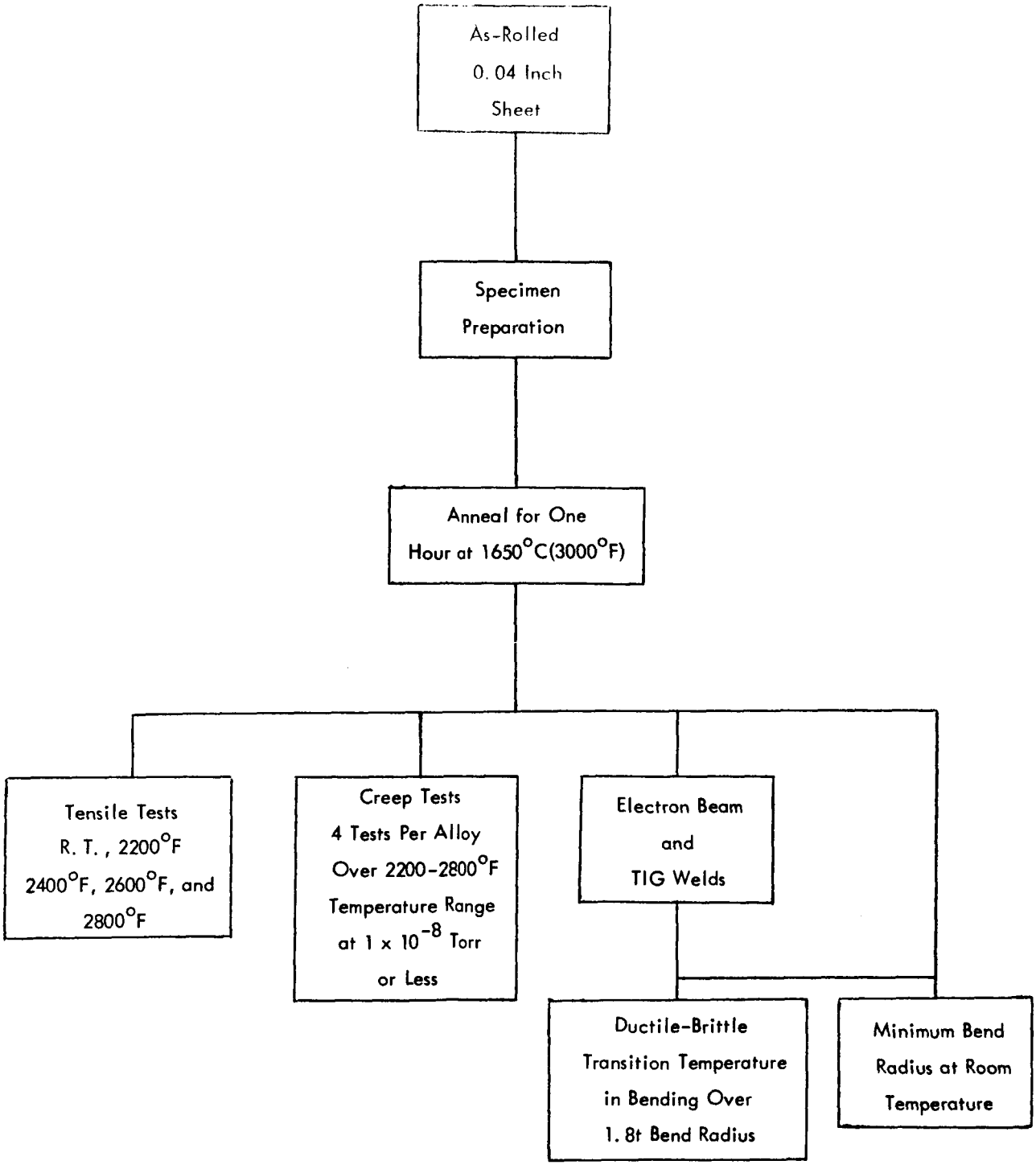


FIGURE 2 - Test Schedule for 0.04 Inch Sheet Processed from Optimized Two Inch Diameter Ingot Compositions

TABLE 1 - Results of Upset Forging of Compositions NASV-12, NASV-13, and NASV-14

Composition (Heat No.)	As-Cast Hardness DPH(30 Kg Load)	Forging Temp. (°C/°F)	Reduction (%)	Upset Constant (K_u)(a) (psi)
Ta-7.5W-1.5Re-0.5Hf-0.015C-0.015N (NASV-12)	336	1300/2372	57	185,000
Ta-6.5W-2.5Re-0.3Zr-0.01C-0.01N (NASV-13	329	1300/2372	67	149,000
Ta-4W-1Mo-2Re-0.3Zr-0.015C-0.015N (NASV-14)	315	1300/2372	59	164,500

(a) $K_u = \frac{E}{VZ}$ where: E = energy used, inch-pounds
V = volume of material deformed, cubic inches
Z = empirical upset factor

TABLE 2 - Creep Results on NASV-14 (Ta-4W-1Mo-2Re-0.3Zr-0.015C-0.015N) at 1315°C (2400°F) and 1 x 10^{-8} Torr

Applied Stress (psi)	Test Duration (hrs.)	Total Plastic Strain (%)	Creep Rate (% per hour)
15,000	230	0.41	0.0018
12,810	211	0.09	0.00043

B. FOUR INCH DIAMETER INGOT SCALE-UP

The composition NASV-20 (Ta-8W-1Re-0.7Hf-0.025C) was selected as a carbide dispersion-strengthened sheet and tubing alloy which should exhibit a good combination of strength, fabricability, and creep resistance based on the results of the Phase I investigation[1]. However, prior to melting as a 4 inch ingot, the cognizant NASA project personnel directed that the composition be first melted as an 800 gram non-consumable melted ingot, processed to 0.04 inch sheet and that weldability and creep strength characteristics be verified.

A summary of room temperature tensile and weld bend transition data are reported in Table 3 for these button heats (NAS-56 and 57). Also in Table 3 are similar data for NASV-9 (Ta-9W-1Hf-0.025C) which show that replacement of 1 w/oW with 1% w/oRe did not significantly change either room temperature tensile properties or weldability characteristics. However, the data plotted in Figure 3 show that the rhenium modification significantly improved the creep resistance at 1315°C (2400°F). After 40 hours of test on the specimen loaded at 15,000 psi, a malfunction in the temperature controller resulted in a temperature disturbance to approximately 2800°F, at which time the test was interrupted by a pressure limiting device. There was no apparent damage to the specimen and thus the test was resumed and continued for an additional 261 hours without further incident.

After verification of the weldability and creep properties of the Ta-8W-1Re-0.7Hf-0.025C, two first melt electrodes for the 4 inch diameter ingots each weighing 42.8 pounds were fabricated using the sandwich construction technique which has been proven successful with the 2 inch diameter ingots. The configuration of the first melt electrodes is shown in Figure 4. First melts are being melted into 2-1/2 inch diameter water cooled copper molds using A. C. vacuum arc-melting practice. Melting data are reported in Table 4 and the first melt ingot is shown in Figure 5. The length limitation of the available 2-1/2 inch diameter molds necessitated that two ingots be produced per electrode.

At the completion of the first melts, the 2-1/2 inch diameter ingots will be assembled to form the second melt electrode which will be melted into a 4 inch diameter ingot using D. C. vacuum arc melting.

C. TURBINE COMPOSITIONS

Two compositions, NASV-10 (Ta-7.1W-1.56Re-0.25Hf-0.12Zr-0.03N) and NASV-11 (Ta-9W-1.5Re-1Hf-0.015Hf-0.015N), selected as material suitable for turbine applications were processed to 3/8 inch diameter rod. Melting and extrusion to round bar were described in the 4th quarterly report[3].

Secondary Working - NASV-10 extruded bar was conditioned, annealed for 1 hour at 1650°C (3000°F) and swaged at 500°C (932°F) to 0.4 inch diameter rod. The total reduction was 76%. NASV-11 extruded bar was conditioned, then coated with Al-12Si, and swaged satisfactorily at 1100°C (2000°F) to 0.4 inch diameter rod for a reduction of 82%.

TABLE 3 - Mechanical Property Data for Ta-8W-1Re-0.7Hf-0.025C (NAS-56 and 57) and Ta-9W-1Hf-0.025C (NASV-9)

	Ta-8W-1Re-0.7Hf-0.025C*	Ta-9W-1Hf-0.025C*
U. T. S. (Room Temperature)	105,000 psi	106,800 psi
Y. S.	78,000	78,600
Elongation		
Uniform	16.7%	14.8%
Total	28	24
Weld Bend Transition Temp.	-100 to -125°F	-125 to -175°F

*Annealed for 1 hour at 1650°C (3000°F) prior to testing.

TABLE 4 - Melting Data for NASV-20

Electrode	Voltage (volts)	A. C. Power (kw)	Melt Rate (lbs/min.)	Ingot Condition
NASV-20 A1	29	95	6.12	Good
NASV-20 A2	28-29	95	6.35	Good
NASV-20 B1	28-29	95-100	6.45	Good

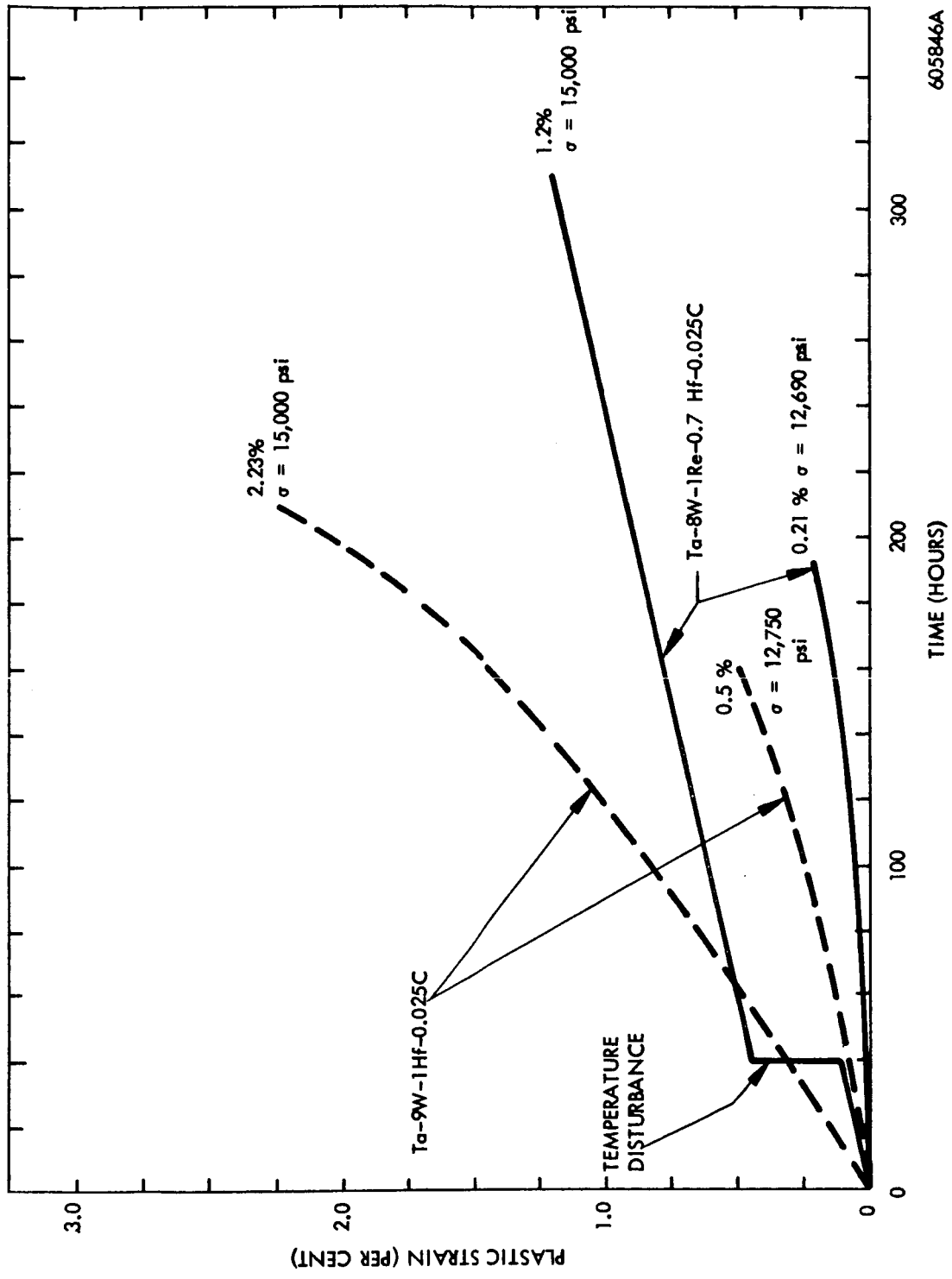


FIGURE 3 - Creep Properties of Ta-9W-0.025C and Ta-8W-1Re-0.7Hf-0.025C Alloys at 1315°C (2400°F) and 1 x 10^{-8} Torr

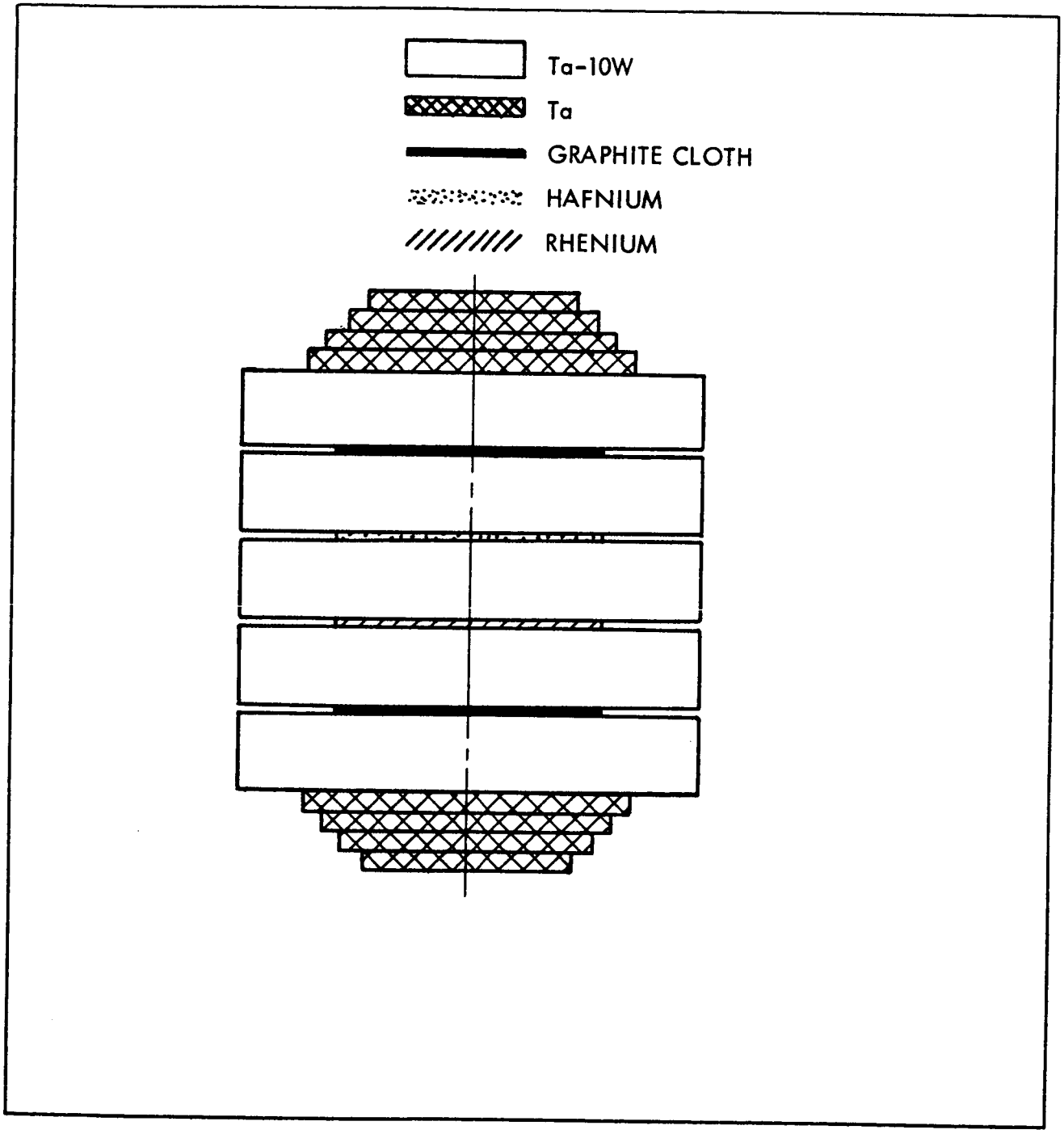


FIGURE 4 - First Melt Electrode Configuration for NASV-20

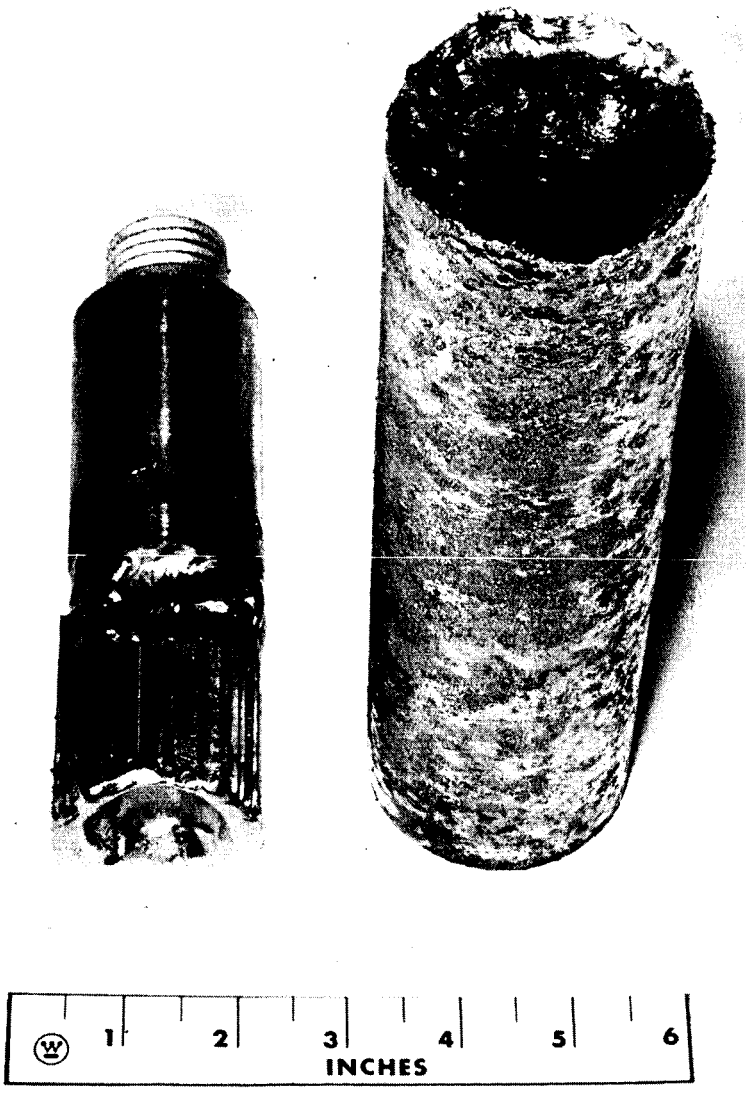


FIGURE 5 - First Melt Ingot, Heat NASV-20

Chemical Analysis - Chemical analysis results obtained on the as-cast ingot for metallics and on as-swaged bar for interstitials are reported below:

	Analysis, weight percent						
	W	Re	Hf	Zr	C	N	O
NASV-10	7.3	1.63	0.28	0.12	0.0029	0.024	0.0022
NASV-11	9.7	1.62	0.92	--	0.016	0.013	0.0063

Recrystallization Temperature - The 1 hour recrystallization temperature was determined on samples cut from the as-swaged rod. The effect of 1 hour at temperature on the microstructure and room temperature hardness is summarized in Table 5. Both alloys formed a single phase equiaxed microstructure after heating for 1 hour at 1600°C (2910°F).

TABLE 5 - Effect on One Hour Annealing Treatment on Microstructure and Room Temperature Hardness of As-Swaged Rod of Compositions NASV-10 & 11

	Room Temperature Hardness[a] and Microstructure[b] After 1 Hour at Temp. (°C/°F)						
Heat No.	As-Swaged	1200/2190	1300/2370	1400/2550	1500/2730	1600/2910	1800/3270
NASV-10	367 W	373 W	380 W	345 W	327 R_p	310 R	310 R
NASV-11	463 W	424 W	390 W	318 R_b	338 R_p	353 R	353 R

(a) Diamond Pyramid Hardness, 30 Kg Load

(b) Microstructure:

W = Wrought

R_b = <50% equiaxed grains

R_p = >50% equiaxed grains

R = >98% equiaxed grains

Mechanical Property Evaluation - Tensile properties for NASV-10 are reported in Table 6. The test specimens were annealed for 1 hour at 1650°C (3000°F) prior to test. For a $K_t = 3$, the notched-unnotched tensile strength ratio at room temperature was 1.5, which is indicative of excellent notch ductility. Creep tests in ultra-high vacuum conditions

for NASV-10 were conducted using round bar test specimens. The round bar grips and test specimen are shown in Figure 6. At 1315°C (2400°F) under an applied stress of 16,600 psi, 15.61% total plastic strain occurred during 140 hours of testing. The minimum creep rate was 0.04% per hour. This same composition non-consumable arc melted during Phase I[1] and designated NAS-39, was processed to 0.04 sheet, annealed 1 hour at 1650°C (3000°F) and then tested at 1315°C (2400°F), elongated 0.2% in 197 hours under an applied stress of 20,000 psi. Chemical analyses showed both compositions were essentially identical although NASV-10 analyzed approximately 60 ppm less nitrogen than NAS-39 (240 vs 300). However, differences in prior thermal mechanical history have been shown[1] to cause wider differences in creep behavior than minor chemistry variations. Metallographic investigation is underway to determine any metallurgical differences existing as a result of variations in prior processing history.

TABLE 6 - Tensile Properties of NASV-10

	0.2% Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%) Uniform	Elongation (%) Total	Reduction In Area (%)
R. T. [(a)]	116,500	129,200	16.8	37.5	83
2000 [(b)]	45,200	66,600	--	28	--
2400 [(b)]	33,600	48,600	--	28	--
3000 [(b)]	23,300	23,500	--	63	--
R. T. (Notched-K_t=3) [(b)]	--	193,000	--	--	67.3

(a) Strain rate 0.005 in/in/min through 0.6% yield and then 0.05 in/in/min for balance of test.

(b) Strain rate of 0.05 in/in/min throughout test.

During the creep test of NASV-10, diffusion bonding occurred between the thoriated tungsten inserts, the specimen, and grips rendering the inserts non-reuseable. Additional thoriated tungsten inserts are being procured and round bar tests will be started again when the new inserts are received.

D. NITROGEN ADDITIONS TO TANTALUM ALLOYS

Chemical analysis results for nitrogen content performed during this period show good agreement with the weight gain measurements which were reported in the 4th Quarterly Progress Report[3]. A comparison of the nitrogen content obtained by weight gain and

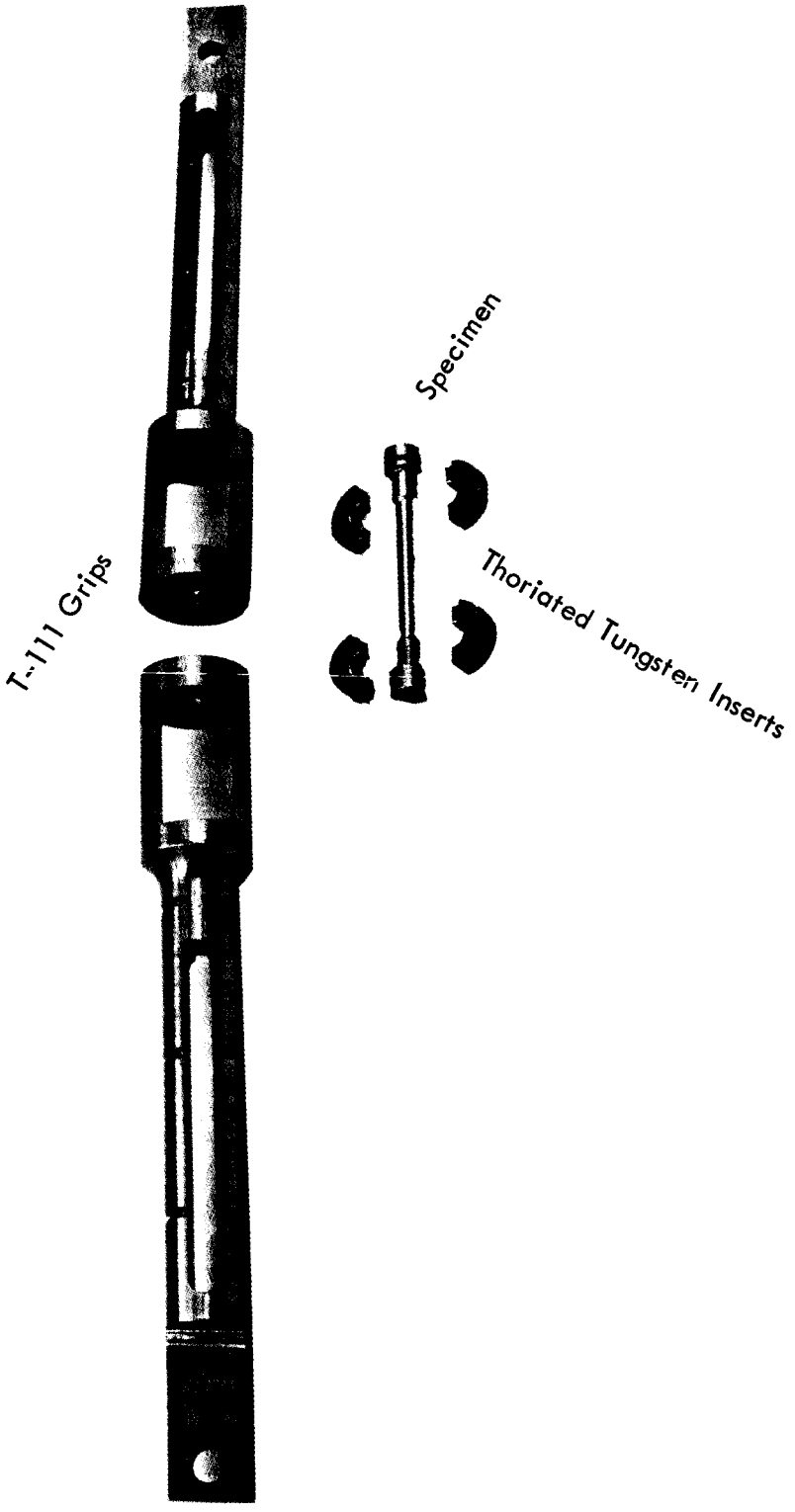


FIGURE 6 - Round Bar Grips and Creep Specimen for Testing in Ultra-High Vacuum

chemical analysis are given below:

Weight Gain	Chemical Analysis
0.17%	0.16%
0.13%	0.12%

Oxygen pickup during nitridation at 1500°C was minimal. Analysis of nitrided tantalum strips containing 0.13% N_2 showed an oxygen content of 0.009%. The ingot from which this strip was processed analyzed 0.005% O_2.

The first 2 inch ingot to which the nitrogen addition was made with nitrided tantalum strips was NASV-11. The addition was 0.015% and the ingot analyzed 0.013% N_2.

E. RESPONSE TO HEAT TREATMENT

1. Effect on Hardness - Thermal treatment investigation of the following compositions was continued.

NAS-36 (Ta-5.7W-1.56Re-0.7Mo-0.25Hf-0.13Zr-0.015C-0.015N)
NAS-39 (Ta-7.1W-1.56Re-0.25Hf-0.13Zr-0.03N)
NAS-42 (Ta-5.3W-0.65Mo-1.56Re-0.52Zr-0.06N)

Samples of each of the above compositions, in various initial metallurgical conditions, were exposed for 500 hours at 1150°C (2100°F) and 1250°C (2280°F). Room temperature hardness measurements taken before and after the thermal treatment are reported in Table 7. Essentially none of the stored energy from the prior cold reduction was retained in the NAS-36 sheet after the 500 hour aging treatment at either temperature. A dispersed second phase was observed in all samples after the aging treatment with a relatively coarse non-continuous grain boundary precipitate observed in the specimens aged at 1150°C (2100°F). This precipitate is presumed to be the dimetal carbide of tantalum and was too large (>5μ) to be an effective dispersion strengthener.

The NAS-39 specimens had essentially the same room temperature hardness after the 1250°C (2280°F) age irrespective of the initial metallurgical condition. A two-phase microstructure with a finely dispersed Zr and/or Hf nitride was observed in the sample aged at 1250°C (2280°F). Nitrogen solubility in pure tantalum has been reported to be 0.44 weight percent at 1200°C (2190°F)[4]. However, the amount of precipitate observed in the microstructure of the aged NAS-39 sheet specimens would certainly indicate that the nitrogen solubility in this tantalum alloy matrix at 1250°C (2280°F) is less than 0.03 weight percent.

A 1 hour aging treatment over the range of 1000-1600°C (1830-2910°F) of solution

TABLE 7 - Room Temperature Hardness[a] of Tantalum Base Alloys After 500 Hours at 1150°C (2100°F) and 1250°C (2280°F) and 10^{-10} Torr

Treatment	Room Temperature Hardness								
	NAS-36[d]			NAS-39[d]			NAS-42[d]		
	Initial Condition	1150°C (2100°F)	1250°C (2280°F)	Initial Condition	1150°C (2100°F)	1250°C (2280°F)	Initial Condition	1150°C (2100°F)	1250°C (2280°F)
A[b]	358	281	253	---	---	---	458	317	275
A + 1 hr. at 1650°C(3000°F) + 2-1/2% C. W.	323	248	257	389	314	287	---	---	---
A + 1 hr. at 1650°C (3000°F) + 5% C. W.	338	260	253	363	329	288	---	---	---
A + 1 hr. at 2000°C(3630°F) + 2-1/2% C. W.	388	285	268	354	307	287	425	355	281
A + 1 hr. at 2000°C(3630°F) + 5% C. W.	362	285	270	379	313	290	438	346	271
As-Forged[c]	---	---	---	---	---	---	474	311	305
As-Forged + 1 hr. at 2000°C (3630°F)	---	---	---	---	---	---	409	421	309

(a) Diamond Pyramid Hardness, 30 Kg Load
(b) A - As-Worked, 0.04 inch thick
(c) Non-consumable electrode melted, 800 gram ingot, upset 50% at 1500°C (2730°F)
(d) See Appendix I for composition

annealed NAS-39 did not cause any significant change in room temperature hardness nor was any precipitate observed when the aged samples were examined metallographically at 1500X. Thus, low nitrogen supersaturation in the tantalum alloy matrix apparently retards the kinetics of the precipitation reaction. After the 1150°C (2100°F), 500 hour age, the hardness values obtained would indicate that part of the stored energy of the prior cold work was retained, although the strengthening contribution from nitride precipitation cannot be separated.

The data plotted in Figure 7 for NAS-42 show that the effect of the prior cold work of the solution annealed material was to increase the precipitation kinetics during the subsequent aging treatments. The effects of cold work on precipitation from a supersaturated solid solution is complex. A number of competing processes, such as rate of zone formation and rate of nucleation and precipitation of an intermediate phase are two mechanisms that are affected by prior cold work. The atomic mismatch between ZrN and the matrix is approximately +39%*. Atomic mismatch between the matrix and the precipitating phase for common alloy precipitation hardening alloy systems is generally on the order of 10% or less[5]. The mechanism of hardening observed in the high nitrogen bearing alloys will have to be investigated in more detail before more meaningful discussion is possible.

Chemical extraction of the precipitates is in process. Identification will be made by x-ray diffraction analysis and particle size and distribution will be determined by electron microscopy.

The changes in properties caused by the various thermal treatment are being followed primarily by indentation hardness measurements. Indentation hardness values, while useful, do not show changes in work hardening rates which does change significantly in age hardening systems. Also, room temperature hardness changes do not necessarily reflect changes in the elevated temperature properties. Heat NAS-49 (Ta-8.1W-0.52Zr-0.08N) responded to solution annealing and aging treatments similar to NAS-42, and maximum room temperature hardness of solution annealed material occurred after aging 1 hour at approximately 1100°C (2100°F), with overaging occurring after 1 hour at 1600°C (2910°F)[3]. NAS-49 sheet, 0.06 inch thick, was solution annealed for 1 hour at 2000°C (3630°F) and cooled rapidly by admitting helium gas into the furnace chamber. A sample of the solution annealed material was then aged for 1 hour at 1100°C (2100°F) to produce maximum hardness while another sample was overaged by heating for 1 hour at 1600°C (2410°F). Changes in elevated temperature properties were determined using hot hardness measurements and the values obtained over the temperature range of R.T. to 1204°C (2200°F) are given in Table 8. The hot hardness values obtained were in the same order as the room temperature hardness with the overaged specimen exhibiting the lowest values at all test temperature. A difference in tensile strength of approximately 20,000 psi at 1204°C (2200°F) is represented by the hardness difference between the aged and overaged specimen. The hardness values obtained on the solution annealed

*With respect to the alloy matrix.

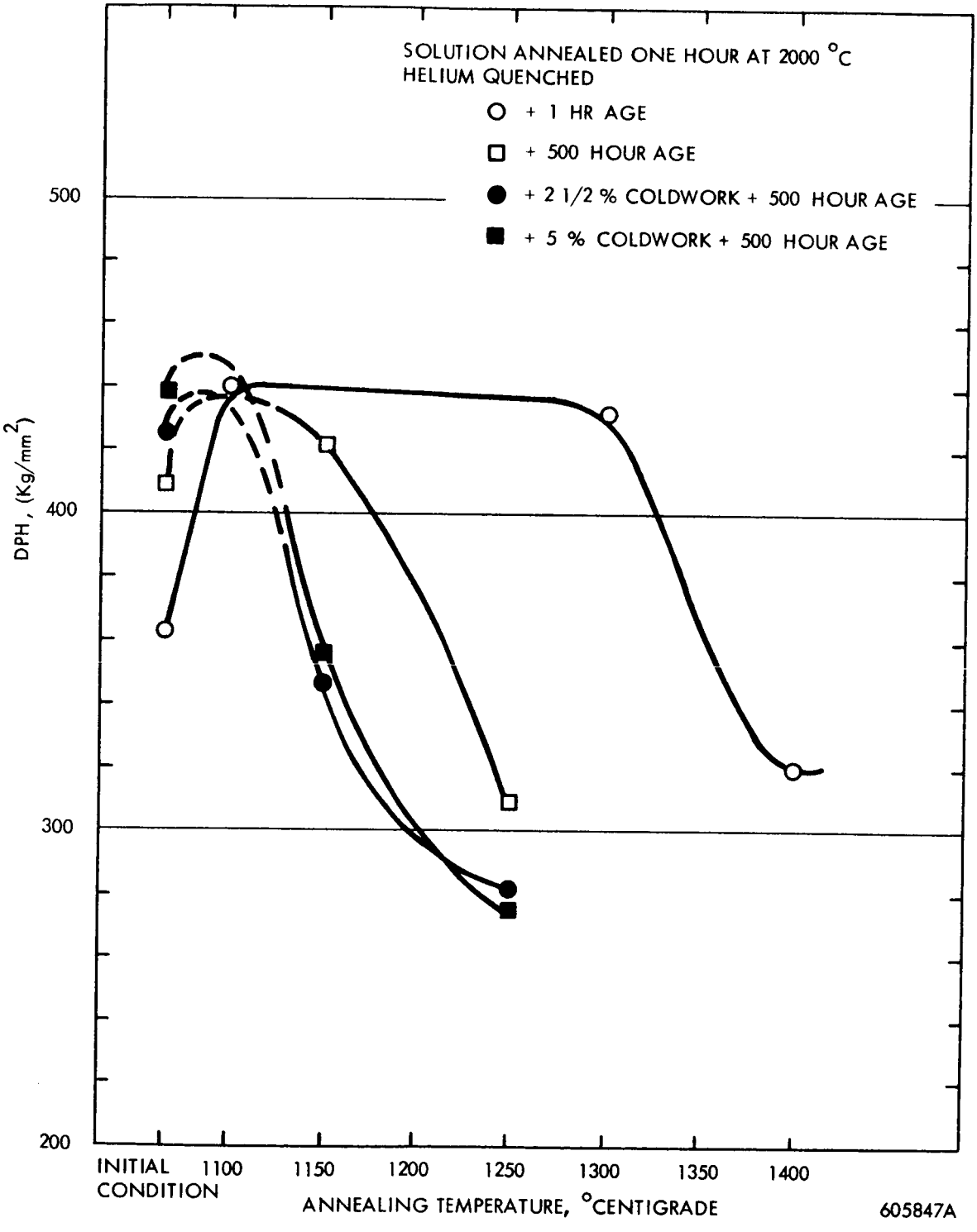


FIGURE 7 - Room Temperature Hardness of NAS-42 (Ta-5.3W-0.65Mo-1.56Re-0.52Zr-0.06N) After Aging for 500 Hours at 10^{-10} Torr

TABLE 8 - Effect of Prior Treatment on the Hot Hardness of Ta-8.1W-0.52Zr-0.08N Alloy

	(DPH at Temperature (°C/°F)				
	R. T.	760/1500	982/1800	1093/2000	1204/2200
A. Annealed 1 hr. at 2000°C, Helium quenched	383	194	171	155	126
B. + 1 hr. at 1100°C	417	217	184	161	149
C. + 1 hr. at 1600°C	365	203	156	141	112

specimen fall between the aged and overaged specimen, as was expected. Aging of the solution annealed specimen during hot hardness testing no doubt occurred, but time at test temperature for all tests were held to a minimum, i.e., 10 minutes or less.

Chemical analyses were made on samples taken from the 500 hour aging treatments at 1250°C (2280°F) and 10^{-10} torr to determine the extent if any of contamination or loss of carbon and/or nitrogen. These results are reported as follows:

Specimen	Pre-Test Analysis	Post-Test Analysis (w/o)
NASV-1(Ta-8W-2Hf)	0.0044 (O_2)	0.0043 (O_2)
NASV-2(Ta-8W-2Hf-0.05C)	0.051 (C)	0.051(C)
NASV-42(Ta-5.3W-1.56Re -0.65Mo-0.52Zr-0.08N)	0.049 (N_2)	0.061 (N_2)

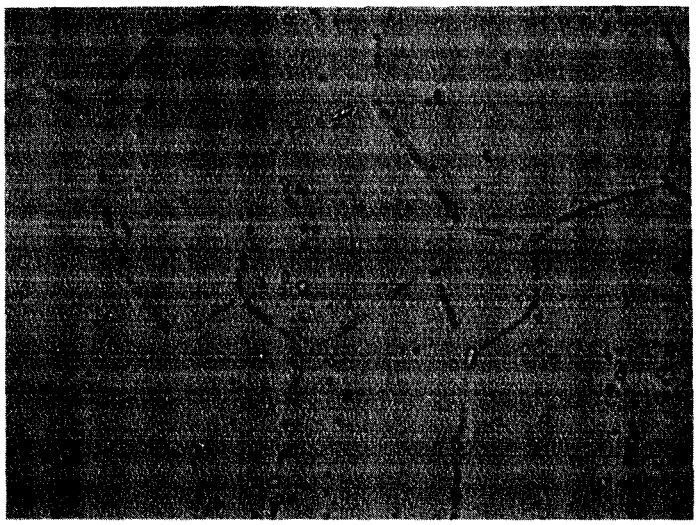
The difference in nitrogen content for NAS-42 is attributed to non-homogeneities in the sheet material which was processed from a non-consumable electrode melted ingot.

2. Effect on Creep Resistance - The initial metallurgical condition has been shown to have a more significant effect on creep resistance than minor compositional variations[1]. Two compositions, NASV-7 (Ta-5.7W-1.56Re-0.7Mo-0.25Hf-0.13Zr-0.015N-0.015C) and NASV-9 (Ta-9W-1Hf-0.025C) were selected to determine the effect of final annealing temperature on the creep resistance at 1315°C (2400°F) and 1×10^{-8} torr under an applied stress of 15,000 psi. The results obtained are summarized in Table 9. Samples were also taken from the gage length of each specimen for metallographic examination. The higher final annealing temperature resulted in an apprent increase in resistance to creep deformation under the conditions of test. The grain size approximately doubled when the final annealing temperature was raised from 1650°C (3000°F) to 1800°C (3270°F). The grain size in both instances is still relatively fine (ASTM-7 or finer) with the NASV-9 having a finer grain size than NASV-7 for the same annealing temperature. Also, metallographic examination at 1500X did not show any apparent difference in the distribution or morphology of the dispersed second phase in the gage section of the test specimen. Examples of the microstructure of NASV-9 are shown in Figure 8. Other than the slight grain size difference the distribution of the second phase particles appear identical.

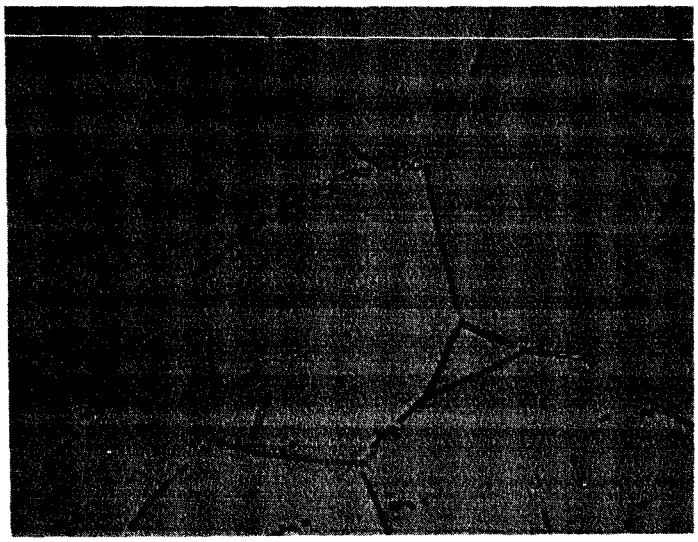
Second phase particles were chemically extracted from the gage length of NASV-7 and NASV-9 test specimens. The bulk extracted residues were identified using x-ray diffraction techniques. X-ray diffraction data reported in Table 10 are for the residues extracted from the specimens annealed one hour at 1650°C (3000°F) prior to creep testing as similar patterns were obtained from the specimens which had been annealed at 1800°C (3270°F) prior to test. Traces of monoclinic HfO_2 were found in all four specimens.

TABLE 9 - Effect of Final Annealing Temperatures on the Creep Resistance of NASV-7 and NASV-9 at 1315°C (2400°F), 15,000 psi and 15^{9} Torr

Specimen	Final Annealing Temperature (°C/°F)	Resulting Average Grain Diameter (mm)	Test Duration (hours)	Total Plastic Strain (per cent)	Time to 1% Plastic Strain (hours)	Minimum Creep Rate (% per hour)
NASV-7 - 1C	1650/3000	0.023	190	0.57	-	0.003
- 5C	1800/3270	0.04	816	4.9	400	0.00125
NASV-9 - 2C	1650/3000	0.014	209	2.2	109	0.0074
- 5C	1800/3270	0.028	500	4.7	180	0.0052



(a) Annealed 1 Hour at 1610°C (3000°F) Test Duration 209 Hours



(b) Annealed 1 Hour at 1800°C (3270°F) Test Duration 501 Hours

FIGURE 8 - Microstructure of NASV-9 Gage Length After Creep Testing at 1315°C 1×10^{-8} Torr and an Applied Stress of 15,000 psi Mag. - 1500X
Etchant - $HF-HNO_3$-Glycerine

TABLE 10 - X-ray Diffraction Data

NASV-9 (Ta-9W-1Hf-0.025C)			NASV-7 (Ta-5.7W-1.56Re-0.7Mo-0.25Hf-0.13Zr-0.015C-0.015N)		
Annealed 1 hour at 1650°C (3000°F) plus 210 hours at 1316°C (2400°F) at Σ = 15,000 and p = 1 x 10^{-8} torr			Annealed 1 hour at 1650°C (3000°F) plus 190 hours at 1316°C (2400°F) at Σ = 15,000 psi and p = 1 x 10^{-8} torr		
d	Intensity	Phase	d	Intensity	Phase
3.15	VVW	HfO_2	3.15	VW	HfO_2
2.95	VVW	N.I.	2.92	W	N.I.
2.82	VVW	HfO_2	2.82	VW	HfO_2
2.68	M	M_2C	2.68	S	M_2C
2.62	W	HfO_2	2.63	M	MC
2.47	M	M_2C	2.52	VVW	HfO_2
2.37	VS	M_2C	2.47	S	M_2C
2.32	VVW	HfO_2	2.36	VS	M_2C
1.98	VVW	HfO_2	2.29	M	MC
1.82	M	M_2C	1.82	MS	M_2C
1.72	VVW	N.I.	1.64	VVW	HfO_2
1.64	VVW	HfO_2	1.62	MW	MC
1.55	M	M_2C	1.55	MS	M_2C
1.41	M	M_2C	1.40	MS	M_2C
1.34	W	M_2C	1.38	W	MC
1.32	M	M_2C	1.34	MW	M_2C
1.30	M	M_2C	1.32	MS	M_2C
1.24	W	M_2C	1.30	M	M_2C
1.18	W	M_2C	1.24	MW	M_2C
1.12	W	M_2C	1.18	MW	M_2C
1.04	MW	M_2C	1.12	MW	M_2C
1.02	W	M_2C	1.05	VVW	N.I.
.997	MS	M_2C	1.04	M	M_2C
.970	MW	M_2C	1.025	VW	M_2C
.942	MW	M_2C	1.015	W	MC
.930	MW	M_2C	.995	MS	M_2C
.912	W	M_2C	.965	M	M_2C
.898	W	M_2C	.940	MW	M_2C
.868	M	M_2C	.928	M	M_2C
.844	MW	M_2C	.910	W	M_2C
.800	W	M_2C	.866	M	M_2C
.788	W	M_2C	.844	M	M_2C
.778	W	M_2C	.797	VW	M_2C

Note:

(a) M_2C is HCP with a_o = 3.12 Å
c_o = 4.96 Å
c/a = 1.593
assumed to be $(Ta,WHf)_2C_{1-x}$

(b) MC is FCC with a_o = 4.60 Å

(c) NI - Not Indexed

Only the HCP dimetal carbide phase, $(Ta, W, Hf)_2 C_{1-x}$, was observed in the residues extracted from the NASV-9 specimens. However, both a HCP phase and a FCC phase were found in the residue from the NASV-7 specimens. The a_o = 3.12 Å and c_o = 4.96 Å for the HCP phase found in both compositions compares very close to that reported for Ta_2C (a_o = 3.106 Å and c_o = 4.945 Å) suggesting that Ta is the primary metallic component of the compound with very minor substitutions of W and/or Hf. The FCC phase found in NASV-7 had a lattice parameter a_o = 4.60 Å which is close to the values reported for $HfC_{.8}$ (4.61 Å) and $ZrN_{.8}$ (4.58 Å). The absence of a FCC monocarbide phase in NASV-9 would indicate that the FCC phase is ZrN with probably some substitution of C for N. There is a dearth of published data on the ZrC-ZrN system, probably because of the instability of the ZrN at temperatures above 2000°C (3630°F). The decrease in creep rate observed when the final annealing temperature was increased can thus far only be attributed to the grain size difference. However, changes in the dispersed phase morphology could also be a possible cause for the observed change in creep rate.

The extended test on NASV-7-5C was conducted to determine if massive grain boundary carbides formed during testing would affect the creep resistance. The shape of the creep curve obtained was typical to that obtained previously[1] and no discontinuities in the creep elongation curve were observed. A section from the gage length of this specimen is also being analyzed by electron beam microprobe techniques to determine if a varying compositional distribution exists across the specimen thickness.

III. FUTURE WORK

During the next quarterly period it is planned to accomplish the following:

1. Complete melting of the optimized 2 inch diameter ingot compositions.
2. Complete melting of the first 4 inch ingot composition NASV-20.
3. Creep testing evaluation under ultra-high vacuum conditions will continue.
4. Phase identification and morphology investigation of dispersed second phases will continue.

IV. REFERENCES

1. R. W. Buckman and R. T. Begley, "Development of Dispersion Strengthened Tantalum Base Alloys," Final Technical Report, Phase I, WANL-PR-(Q)-004.

2. R. L. Ammon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet," Summary Phase Report WANL-PR-M-004, June 15, 1963, Prepared under Navy, Bureau of Naval Weapons, Contract NOw-62-0656-d.

3. R. W. Buckman, "Development of Dispersion Strengthened Tantalum Base Alloy," 4th Quarterly Report, WANL-PR-(Q)-005.

4. G. Bauer and K. H. Zapp, "Z Anorg. Chem." 277, 129 (1954).

5. A. Kelley and R. B. Nicholson, "Precipitation Hardening," Progress in Metals Physics, Volume 10, No. 3, 1963, Pages 286 and 287.

APPENDIX I

Compositions of alloys discussed in this report are listed below:

Heat Number	Composition Weight Per cent
	Consumable Electrode Melted
NASV-7	Ta-5.7W-1.56Re-0.7Mo-0.25Hf-0.13Zr-0.015N-0.015C
NASV-9	Ta-9W-1Hf-0.025C
NASV-10	Ta-7.1W-1.56Re-0.25Hf-0.12Zr-0.03N
NASV-11	Ta-9W-1.5Re-1Hf-0.015C-0.015N
NASV-12	Ta-7.5W-1.5Re-0.5Hf-0.015C-0.015N
NASV-13	Ta-6.5W-2.5Re-0.3Hf-0.01C-0.01N
NASV-14	Ta-4W-1Mo-2Re-0.3Zr-0.015C-0.015N
NASV-15	Ta-9W-1.5Re-1Hf-0.06N
NASV-16	Ta-9.5W-0.5Re-0.25Zr-0.02C-0.01N
NASV-17	Ta-4W-3Re-0.75Hf-0.01C-0.02N
NASV-18	Ta-6W-1Mo-1Re-0.7Hf-0.025C
NASV-20	Ta-8W-1Re-0.7Hf-0.025C
	Non-Consumable Electrode Melted
NAS-36	Ta-5.7W-1.56Re-0.7Mo-0.25Hf-0.13Zr-0.015C-0.015N
NAS-39	Ta-7.1W-1.56Re-0.25Hf-0.13Zr-0.03N
NAS-42	Ta-5.3W-0.65Mo-1.56Re-0.52Zr-0.06N
NAS-56, 57	Ta-8W-1Re-0.7Hf-0.025C

DISTRIBUTION LIST

TRW
Caldwell Research Center
23555 Euclid Avenue
Cleveland, Ohio, 44117
Attn: Librarian
G. J. Guarnieri

TRW
New Devices Laboratories
7209 Platt Avenue
Cleveland, Ohio 44104
Attn: Librarian

TRW
Space Technology Laboratories
One Space Park
Redondo Beach, California
Attn: Librarian

National Aeronautics & Space Adm.
Washington, D. C. 20546
Attn: Walter C. Scott
James J. Lynch (RN)
George C. Deutsch (RR)

National Aeronautics & Space Adm.
Scientific & Technical Inf. Facility
Box 5700
Bethesda, Maryland 21811

National Aeronautics & Space Adm.
Ames Research Center
Moffet Field, California 94035
Attn: Librarian

National Aeronautics & Space Adm.
Goddard Space Flight Center
Greenbelt, Maryland 20771
Attn: Librarian

National Aeronautics & Space Adm.
Langley Research Center
Hampton, Virginia 23365
Attn: Librarian

National Aeronautics & Space Adm.
Manned Spacecraft Center
Houston, Texas 77001
Attn: Librarian

National Aeron tics & Space Adm.
George C. Mars ll Space Flight Center
Huntsville, Al ama 35812
Attn: Librari
Wm. A. lson R-ME-MM

National Aeron tics & Space Adm.
Jet Propulsion boratory
4800 Oak Grove ive
Pasadena, Cali nia 91103

National Aeron tics & Space Adm.
21000 Brookpar oad
Cleveland, Ohi 4135
Attn: Librari
Dr. Ber d Lubarsky (SPSD)
Roger M er (NPTB)
G. M. A
Joe Joy (NPTB)
Paul Moorhead (NPTB)
John J. Fackler (SPSPS)
Norman T. Musial
Thomas Strom
T. A. Moss (NPTB)
Dr. Louis Rosenblum
John Creagh
G. K. Watson
Tom Moore
George Tulsiak
Report Control Section

DISTRIBUTION LIST
(continued)

National Aeronautics & Space Adm.
Western Operations Office
150 Pico Boulevard
Santa Monica, California 90406
Attn: Mr. John Keeler

National Bureau of Standards
Washington 25, D. C.
Attn: Librarian

Aeronautical Systems Division
Wright-Patterson Air Force Base
Ohio
Attn: Charles Armbruster (ASRPP-10)
T. Cooper
Librarian
John L. Morris
H. J. Middendorp (ASNRG)
George Thompson (APIP-1)
George Sherman (API)

Army Ordnance Frankford Arsenal
Bridesburg Station
Philadelphia 37, Pennsylvania
Attn: Librarian

Bureau of Ships
Dept. of the Navy
Washington 25, D. C.
Attn: Librarian

Bureau of Weapons
Research and Engineering
Material Division
Washington 25, D. C.
Attn: Librarian

U. S. Atomic Energy Commission
Technical Reports Library
Washington 25, D. C.
Attn: J. M. O'Leary

U. S. Atomic Energy Commission
P. O. Box 1102
East Hartford, Connecticut
Attn: C. E. McColley
CANEL Project Office

U. S. Atomic Energy Commission
Germantown, Maryland
Attn: Col. E. L. Douthett
SNAP 50/SPUR Project Office

H. Rochen
SNAP 50/SPUR Project Office

Socrates Christopher

Major Gordon Dicker
SNAP 50/SPUR Project Office

U. S. Atomic Energy Commission
Technical Information Service Extension
P. O. Box 62
Oak Ridge, Tennessee

U. S. Atomic Energy Commission
Washington 25, D. C.
Attn: M. J. Whitman

Argonne National Laboratory
9700 South Cross Avenue
Argonne, Illinois
Attn: Librarian

Brookhaven National Laboratory
Upton, Long Island, New York
Attn: Librarian

Oak Ridge National Laboratory
Oak Ridge, Tennessee
Attn: W. C. Thurber
Dr. A. J. Miller
Librarian

Office of Naval Research
Power Division
Washington 25, D. C.
Attn: Librarian

U. S. Naval Research Laboratory
Washington 25, D. C.
Attn: Librarian

DISTRIBUTION LIST
(continued)

Advanced Technology Laboratories
Division of American Standard
369 Whisman Road
Mountain View, California
Attn: Librarian

Aerojet General Corporation
P. O. Box 296
Azusa, California
Attn: Librarian

Aerojet General Nucleonics
P. O. Box 77
San Ramon, California
Attn: Librarian

AiResearch Manufacturing Co.
Sky Harbor Airport
402 South 36th Street
Phoenix, Arizona
Attn: Librarian
E. A. Kovacevich
John Dannan

AiResearch Manufacturing Co.
9851-9951 Sepulveda Boulevard
Los Angeles 45, California
Attn: Librarian

I. I. T. Research Institute
10 W. 35th Street
Chicago, Illinois 60616

Atomics International
8900 DeSoto Avenue
Canoga Park, California

Avco
Research & Advanced Development Dept.
201 Lowell Street
Wilmington, Massachusetts
Attn: Librarian

Babcock and Wilcox Co.
Research Center
Alliance, Ohio
Attn: Librarian

Battelle Memorial Institute
505 King Avenue
Columbus, Ohio
Attn: C. M. Allen
Librarian

The Bendix Corporation
Research Laboratories Div.
Southfield, Detroit 1, Michigan
Attn: Librarian

The Boeing Company
Seattle, Washington
Attn: Librarian

Brush Beryllium Company
Cleveland, Ohio
Attn: Librarian

Carborundum Company
Niagara Falls, New York
Attn: Librarian

Chance Vought Aircraft, Inc.
P. O. Box 5907
Dallas 22, Texas
Attn: Librarian

Clevite Corporation
Mechanical Research Division
540 East 105th Street
Cleveland 8, Ohio
Attn: Mr. N. C. Beerli
Project Administrator

Climax Molybdenum Co. of Michigan
Detroit, Michigan
Attn: Librarian

Convair Astronautics
5001 Kerrny Villa Road
San Diego 11, California
Attn: Librarian

E. I. duPont de Nemours & Co., Inc.
Wilmington 98, Delaware
Attn: Librarian

DISTRIBUTION LIST
(continued)

Electro-Optical Systems, Inc.
Advanced Power Systems Division
Pasadena, California
Attn: Librarian

Fansteel Metallurgical, Corp.
North Chicago, Illinois
Attn: Librarian

Ford Motor Company
Aeronutronics
Newport Beach, California
Attn: Librarian

General Dynamics/General Atomic
P. O. Box 608
San Diego, California 92112
Attn: Librarian

General Electric Company
Atomic Power Equipment Div.
P. O. Box 1131
San Jose, California

General Electric Company
Flight Propulsion Laboratory Dept.
Cincinnati 15, Ohio
Attn: Librarian

General Electric Company
Missile and Space Vehicle Dept.
3198 Chestnut Street
Philadelphia 4, Pennsylvania
Attn: Librarian

General Electric Company
Vallecitos
Vallecitos Atomic Lab.
Pleasanton, California
Attn: Librarian

General Electric Company
Re-Entry Systems Dept.
Cincinnati, Ohio
Attn: Dr. J. W. Semmel

General Electric
Lamp Metals & Components Dept.
21800 Tungsten Road
Cleveland, Ohio
Attn: Librarian

General Dynamics/Fort Worth
P. O. Box 748
Fort Worth, Texas
Attn: Librarian

General Motors Corp.
Allison Division
Indianapolis 6, Indiana
Attn: Librarian

Hamilton Standard
Div. of United Aircraft Corp.
Windsor Locks, Connecticut
Attn: Librarian

Hughs Aircraft Company
Engineering Division
Culver City, California
Attn: Librarian

Lockheed Missiles and Space Div.
Lockheed Aircraft Corp.
Sunnyvale, California
Attn: Librarian
Mr. John N. Cox

Marquardt Aircraft Co.
P. O. Box 2013
Van Nuys, California
Attn: Librarian

The Martin Company
Baltimore 3, Maryland
Attn: Librarian

The Martin Company
Nuclear Division
P. O. Box 5042
Baltimore 20, Maryland
Attn: Librarian

Martin Marietta Corp.
Metals Technology Laboratory
Wheeling, Illinois

Massachusetts Institute of Technology
Cambridge 39, Massachusetts
Attn: Librarian

DISTRIBUTION LIST
(continued)

Materials Research & Development
Manlabs, Inc.
21 Erie Street
Cambridge 39, Massachusetts

Materials Research Corp.
Orangeburg, New York
Attn: Librarian

McDonnell Aircraft
St. Louis, Missouri
Attn: Librarian

North American Aviation
Los Angeles Division
Los Angeles 9, California
Attn: Librarian

Pratt & Whitney Aircraft
400 Main Street
East Hartford 8, Connecticut
Attn: Librarian

Norton Company
Worcester, Massachusetts
Attn: Librarian

Pratt & Whitney
Plant N
North Haven, Connecticut
Attn: Librarian

Pratt & Whitney Aircraft
CANEL
P. O. Box 611
Middletown, Connecticut
Attn: Librarian
Dr. Robert Strouth

Republic Aviation Corporation
Farmingdale, Long Island, New York
Attn: Librarian

Solar
2200 Pacific Highway
San Diego 12, California
Attn: Librarian

National Research Corp.
45 Industrial Place
Cambridge, Massachusetts
Attn: R. W. Douglas

Southwest Research Institute
8500 Culebra Road
San Antonio 6, Texas
Attn: Librarian

Rocketdyne
Canoga Park, California
Attn: Librarian

Superior Tube Company
Norristown, Pennsylvania
Attn: Mr. A. Bound

Sylvania Electric Products, Inc.
Chem. & Metallurgical
Towanda, Pennsylvania
Attn: Librarian

Temescal Metallurgical
Berkeley, California
Attn: Librarian

Union Carbide Metals
Niagara Falls, New York
Attn: Librarian

Union Carbide Stellite Corp.
Kokomo, Indiana
Attn: Librarian

Union Carbide Nuclear Co.
P. O. Box X
Oak Ridge, Tennessee
Attn: X-10 Laboratory Records Dept.

United Nuclear Corp.
5 New Street
White Plains, New York
Attn: Librarian
Mr. Albert Weinstein,
Senior Engineer

Universal Cyclops Steel Corp.
Refractomet Division
Bridgeville, Pennsylvania
Attn: C. P. Mueller

University of Michigan
Department of Chemical & Metallurgical Eng.
Ann Arbor, Michigan
Attn: Librarian

DISTRIBUTION LIST
(continued)

Vought Astronautics
P. O. Box 5907
Dallas 22, Texas
Attn: Librarian

Wah Chang Corp.
Albany, Oregon
Attn: Librarian

Wolverine Tube Division
Calumet & Hecla, Inc.
17200 Southfield Road
Allen Park, Michigan
Attn: R. C. Cash

Wyman-Gordon Company
North Grafton, Massachusetts
Attn: Librarian