WANL-PR(Q)-012
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DEVELOPMENT OF DISPERSION STRENGTHENED TANTALUM BASE ALLOY

Eleventh Quarterly Report

by

R. W. Buckman and R. C. Goodspeed

prepared for
National Aeronautics and Space Administration
Lewis Research Center
Space Power Systems Division
Under Contract (NAS 3-2542)

ASTRONUCLEAR LABORATORY
WESTINGHOUSE ELECTRIC CORPORATION

Astronuclear Laboratory

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ELEVENTH QUARTERLY PROGRESS REPORT

Covering the Period

May 20, 1966 to August 20, 1966

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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Technical Management
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ABSTRACT

Development of dispersion strengthened tantalum base alloys for use in advanced space power systems continued with the composition selection and melting of scale-up compositions Ta-8W-1Re-1Hf (ASTAR-811) and Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN) as 4 inch diameter ingots. The creep behavior of Ta-8W-1Re-0.7Hf-0.025C (ASTAR-811C) was affected by final annealing temperature with time to 1% strain at 2400°F and 15,000 psi increasing from 262 to 494 hours as the 1 hour pretest annealing temperature was increased from 3000°F to 3630°F. The time to 1% strain for TIG welds tested in the as-welded condition ut 2400°F and 15,000 psi was 171 hours.

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I. INTRODUCTION

This, the eleventh quarterly progress report on the NASA-sponsored program "Development of a Dispersion Strengthened Tantalum Base Alloy", describes the work accomplished during the period May 20 to August 20, 1966. The work was performed under Contract NAS 3-2542.

The primary objective of the current phase of this program is the double vacuum arc melting of three compositions in the form of 60-pound, 4-inch diameter ingots. These compositions are to be selected for potential sheet and tubing application on the basis of their weldability, creep resistance, and fabricability characteristics.

Prior to this quarterly period, several promising tantalum alloy compositions were developed! These alloys exhibited good creep resistance at 1315°C (2400°F) while maintaining adequate fabricability. From these alloys, a weldable composition containing a carbide dispersion, Ta-8W-0.7Hf-1Re-0.025C (ASTAR-811C) was selected and melted as the first 4-inch diameter ingot (Heat NASV-20). The bottom portion of this ingot was upset forged and processed to 0.04 inch sheet. Another section was side forged and processed to 0.04 inch sheet and detailed information on processing characteristics was obtained[5].

During this quarterly period, the evaluation of the Ta-8W-0.7Hf-1Re-0.025C (ASTAR-811C) composition was essentially completed with the exception of a few remaining creep tests. Composition Ta-8W-1Re-1Hf (ASTAR-811) a solid solution strengthened alloy, and Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN) a carbonitride strengthened composition were both melted as 4-inch diameter ingots and processing to sheet was initiated. This completed melting of the scale-up compositions.

The evaluation of the creep behavior of composition Ta-8W-1Re-0.7Hf-0.025C (ASTAR-811C), heat NASV-20, continued. The creep resistance of as-TIG welded material was significantly less than the base metal.

II. PROGRAM STATUS

A. SCALE-UP

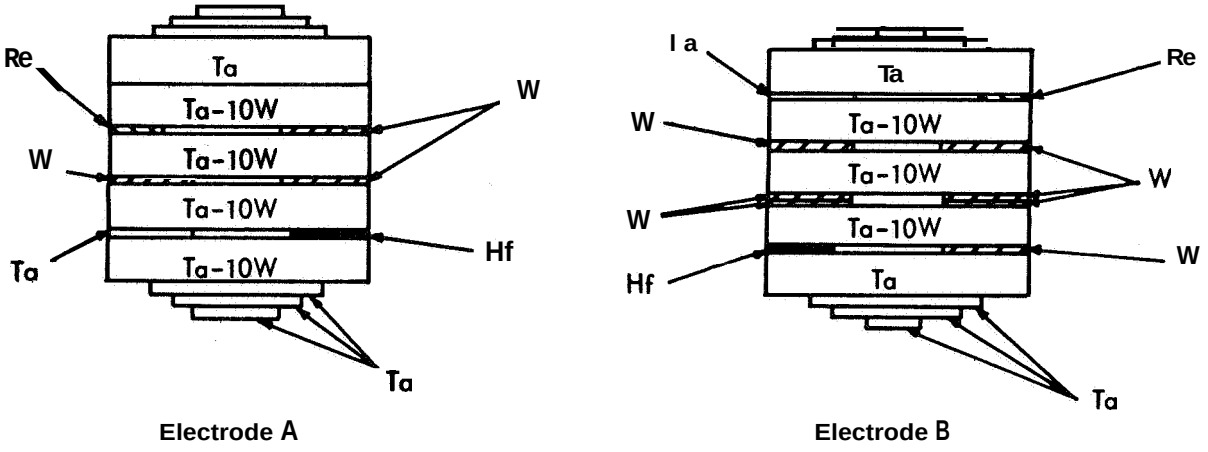
During this period, authorization[2] was received from the cognizant NASA-project manager to proceed with the melting of the two remaining 4-inch diameter ingots. The compositions selected were:

Ta-8W-1Re-1Hf	(ASTAR-811)
Ta-7W-1Re-1Hf-0.012C-0.012N	(ASTAR-811CN)

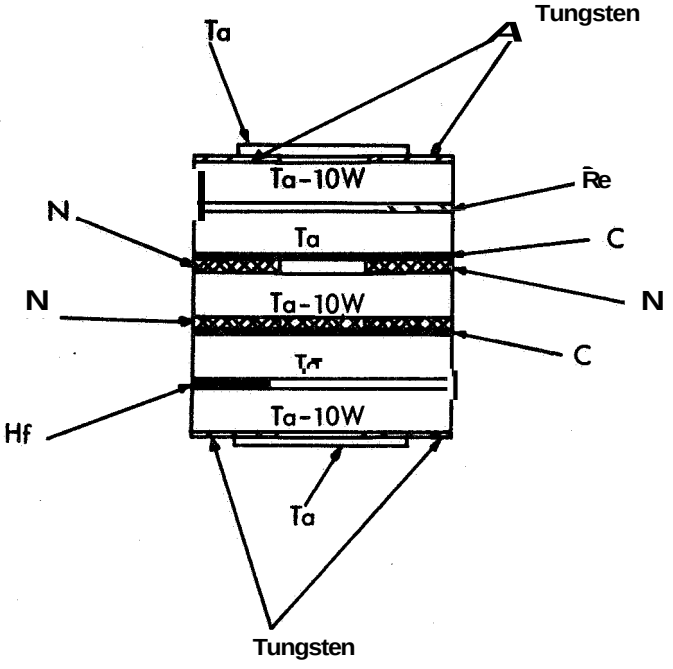
and are representative of solid solution strengthened and solid solution plus carbonitride strengthened alloys[3]. The solid solution strengthened alloy composition has been designated ASTAR-811 and the 4-inch ingot was assigned heat number NASV-22. The solid solution plus carbonitride strengthened composition has been designated ASTAR-811CN and the 4-inch ingot was assigned heat number NASV-23. The carbide strengthened composition Ta-8W-1Re-0.7Hf-0.025C (ASTAR-811C) was melted as the first scale-up ingot and evaluation of this composition has been essentially completed.

1. Melting – Two first melt electrodes, each weighing approximately 40 pounds were required for each heat. Sandwich type electrodes were fabricated from elemental metal strip. A cross-sectional view for each of the first melt electrodes is illustrated in Figure 1. The average cross-section composition of each electrode was at the nominal value for each constituent with the exception of the nitrogen addition to NASV-23. Loss of nitrogen during melting had been observed previously[4]. Thus approximately 30% excess nitrogen was added to the first melt electrode to make up for the anticipated loss during melting.

The first melt electrodes were cast into a water cooled 2-1/2 inch diameter copper mold using AC power. A total of four first melt ingots were produced for each heat. The ends of the first melt ingots were faced, connected by studs, and welded to form the second melt electrode[6]. Final melting was into a 4-inch diameter mold using DC power. First and second melt data for heats NASV-22 and 23 are listed in Table 1.



ASTAR-811
NASV-22 (Average Cross-Section Composition Ta-8W-1Re-1Hf)



ASTAR-811CN
NASV-23 (Average Cross-Section Composition Ta-8W-1Re-0.95Hf-0.012C-0.016N)
(Carbon Added as Graphite Cloth and Nitrogen with Nitrided Tantalum Strip)

FIGURE 1 - Cross-Sectional Make-up of First Melt Electrodes

TABLE 1 - Melting Data for NASV-22 (ASTAR-811) and NAS-23 (ASTAR-811CN)

Melt No.	Electrode Identification	Melting				Ingot Data	
		Volts (AC)	Current (amps)	Melt Rate (lbs/min)	Chamber Pressure (torr)	Dia. (in.)	Weight (lbs.)
ASTAR-811							
CVAM-208	NASV-22A-1	29	3,150	6.4	$<5 \times 10^{-4}$	2.5	23.6
CVAM-209	NASV-22A-2	29	3,150	4.4	a $\times 10^{-4}$	2.5	18.6
CVAM-210	NASV-22B-1	29	3,150	6.9	$<5 \times 10^{-4}$	2.5	24. 1
CVAM-211	NASV-22B-2	29	3,150	6.8	$<5 \times 10^{-4}$	2.5	17.6
CVAM-216	NASV-22	30(DC)	5,500(DC)	3.1 (b)	$<5 \times 10^{-4}$	4.0	69.7
ASTAR-811CN							
CVAM-212	NASV-23B-1	29	3,150	6.5	$<5 \times 10^{-4}$	2.5	23.4
CVAM-213	NASV-23B-2	29	3,150	5.9	$<5 \times 10^{-4}$	2.5	16. 4
CVAM-214	NASV-23A-1	29	3,150	6.2	$<5 \times 10^{-4}$	2.5	23.6
CVAM-215	NASV-23A-2	29	3,150	6.2	$<5 \times 10^{-4}$	2.5	17. 2
CVAM-217	NASV-23	30(DC)	5,500 (DC)	--	$<5 \times 10^{-4}$	4.0	55. 0(a)

(a) Conditioned ingot weight with hot top removed.

(b) Based on total melt duration which includes time for hot topping.

Samples were removed from the top and bottom of each ingot and analyzed for the metallic and interstitial additions. The chemical analysis results on the second melt ingots in Table 2 are indicative of the excellent compositional control obtained with the melting practice used.

TABLE 2 - Chemical Analysis Results for Heats NASV-22 and 23

Nominal Composition (w/o)	Ingot Location	Chemical Analysis, weight percent					
		W	Hf	Re	C	N	O
Ta-8W-1Re-1Hf(NASV-22)	Top	7.8	0.95	1.04	0.0009	0.0010	0.0012
	Bottom	7.5	1.06	0.99	0.0016	0.0013	0.0020
Ta-7W-1Re-1Hf-0.012C-0.012N(NASV-23)	Top	6.5	0.98	1.05	0.013	0.013	0.0018
	Bottom	6.4	1.01	1.01	0.011	0.011	0.0018

Each ingot was lathe conditioned and the hot-top and starting pads were removed. A 1 inch slice was then cut from the bottom portion of the ingot. The ingots after conditioning are shown in Figure 2. The conditioned weight of NASV-22 was 61 pounds and NASV-23, 55 pounds, representing yields from the first melt electrode of 72% and 68% respectively.

2. Ingot Microstructure - A sample removed from the bottom of each ingot was mounted and examined metallographically. Photomicrographs of the as-cast microstructure are shown in Figures 3 and 4. The microstructure of the Ta-8W-1Hf-1Re (NASV-22) is essentially single phase with a minor amount of grain boundary precipitate observable. This precipitate is most probably HfO_2. The microstructure of the carbonitride strengthened composition, Ta-7W-1Re-1Hf-0.012C-0.012N (NASV-23) contains a well defined dispersed second phase which is most likely the dimetal carbide (Ta_2C). The identity of this precipitate is being confirmed. The as-cast hardness of NASV-22 was 231 DPH while that of NASV-23 was 300 DPH.

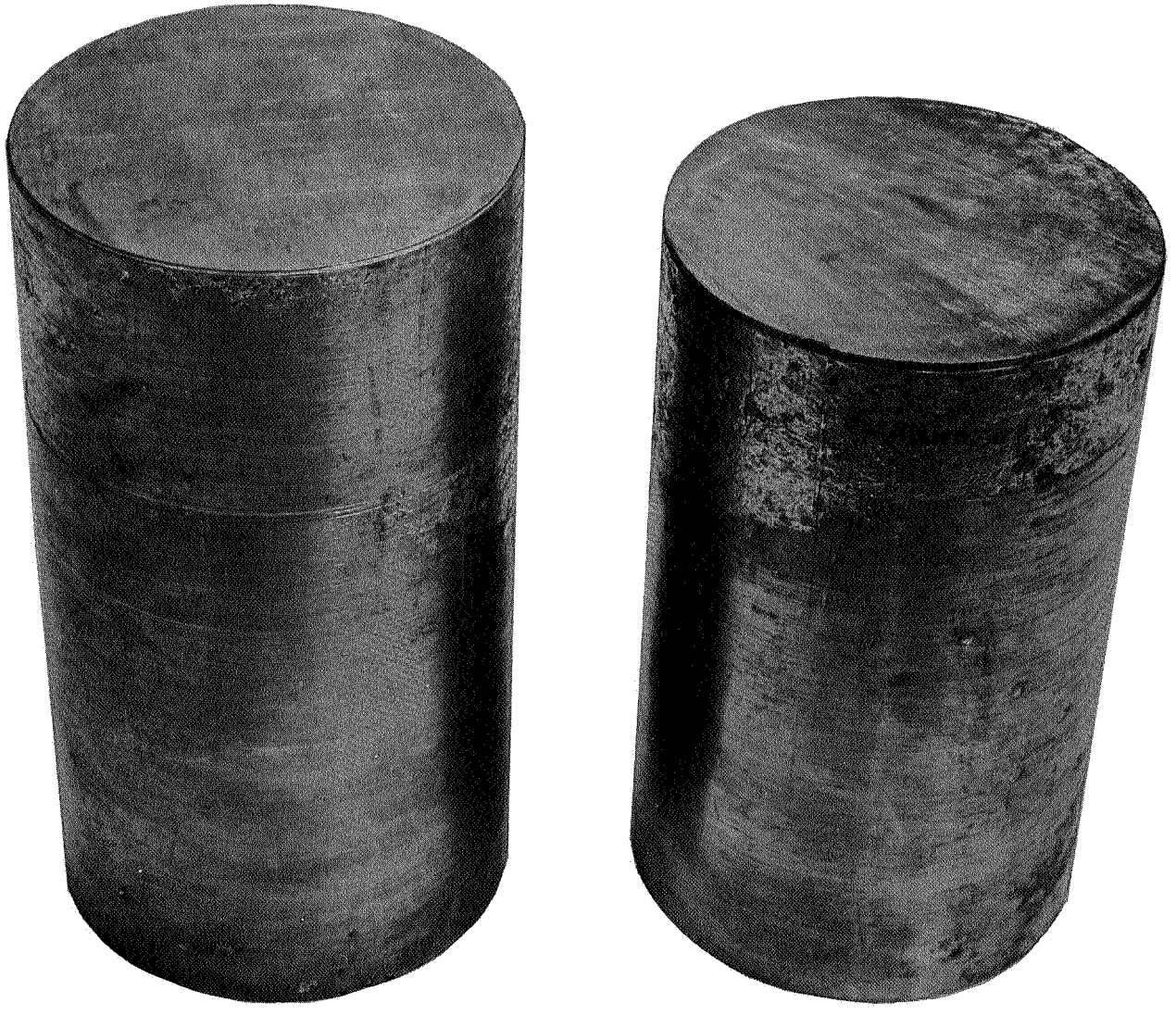
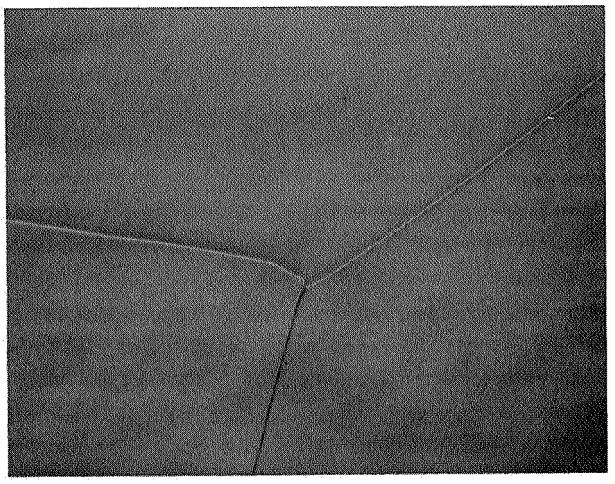
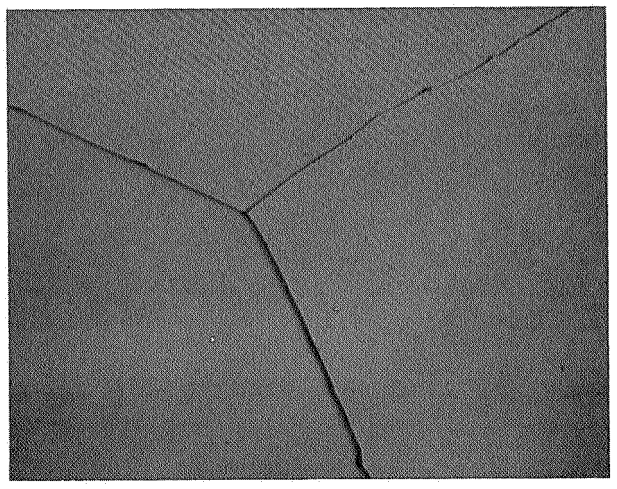


FIGURE 2 - Conditioned Four Inch Diameter Consumable Electrode Double Vacuum Arc Melted Ta Alloy Ingots

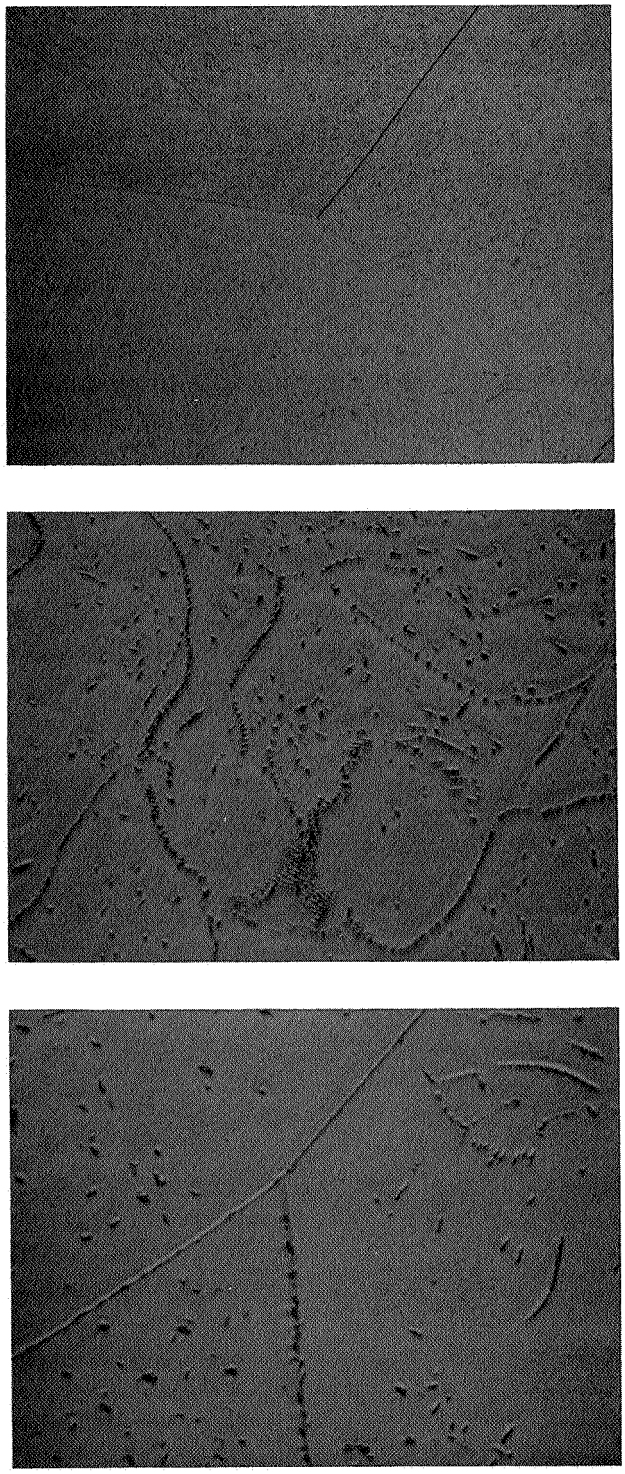


a. 150X



b. 500X

FIGURE 3 - Microstructure of As-Cast Ta-8W-1Re-1Hf (ASTAR-811) Heat NASV-22 Etchant (25% HNO_3 - 25% HF - 50% Glycerine) Oblique Lighting



a. 150X

b. 500X

c. 1500X

FIGURE 4 - Microstructure of As-Cast Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN) Heat NASV-23. Etchant (25%, HNO_3-25% HF-50% Glycerine). Oblique Lighting

3. Primary Working – Four inch diameter x one inch thick billets, each weighing 6-1/2 pounds, from NASV-22 and 23 were coated with Al-12Si and then upset forged at 1400°C in a single blow on the Dynapak. The forging data are in Table 3. Both compositions exhibited excellent forgeability characteristics. There were no defects observed on either of the as-forged billets. The as-forged billets are being conditioned and will then be annealed at 1650°C (3000°F) for 1 hour prior to processing to sheet. The processing schedule of the NASV-22 and 23 will be the same as that used for ASTAR-811C (Heat NASV-20)(5).

4. Mechanical Property Evaluation

a. ASTAR-811C (Heat NASV-20) – The creep behavior of heat NASV-20 of composition Ta-8W-1Re-0.7Hf-0.025C (ASTAR-811C) has been investigated over the temperature range of 2200-2800°F. The data accumulated thus far are listed in Table 4 and plotted in Figure 5 using the Larson-Miller parameter. Increasing the final annealing temperature results in an increase in creep resistance which has been shown to follow a functional relationship of the square root of grain diameter(3). The recrystallized grain diameter is also affected by the prior mechanical history. For sheet material annealed 1 hour at 3000°F, the recrystallized grain diameter increased from 0.017 mm for sheet reduced 83% before annealing to 0.033 mm for a prior reduction of 33%(3). TIG welding also caused a significant decrease in the 2400°F creep behavior. The time to elongate 1% at 2400°F and 15,000 psi for the TIG weld was 171 hours compared to 262 hours for the base material. The two tests listed in Table 4 are stilt in progress and will be continued until rupture so that the fracture behavior can be studied.

III. FUTURE WORK

During the next quarterly period, we plan to accomplish the following:

1. Process the upset forged billets of NASV-22 and NASV-23 to 0.04 inch sheet and initiate mechanical property evaluation.
2. Initiate response-to-heat treatment investigation of NASV-22 and NASV-23.
3. Complete creep property evaluation of ASTAR-811C (Heat NASV-20).

TABLE 3 - Forging Results for Compositions Ta-8W-1Re-1Hf (NASV-22) and Ta-7W-1Re-1Hf-0.012C-0.012N (NASV-23)

Forging Billet				As Forged Billet			
Heat No.	Dimensions Dia. (in.)	Height (in.)	Hardness (DPH)	Forging Temperature (°C/°F)	Dia. (in.)	Height (in.)	Hardness (DPH)	Upset (%)
NASV-22	4	1	231	1400/2550	5-1/4	0.44	292	56
NASV-23	4	1	300	1400/2550	5-1/4	0.44	355	56

TABLE 4 - Creep Results for ASTAR-811C, Ta-8W-1Re-0.7Hf-0.025C (Heat NASV-20)

Temperature (°F)	Pre-Test Annealing Temp. (°F)	Recrystallized Grain Size (mm)	Stress (psi)	Test Duration (hours)	Elongation (%)	Time to Elongate 1% (hours)	Hardness (DPH)		
							Pre-Test	Post-Test	
								Head Section	Gage Length
2400	3000	0.033	12,500	531	0.93	570(a)	255	---	---
2400	3000	0.033	15,000	554(c)	2.53	262	249	229	---
2400	3000	0.017	15,000	457(c)	3.0	202		---	---
2200	3000	0.017	18,000	1,017	1.0	1,017(a)	255	218	217
2600	3000	0.017	8,000	165	0.88	188	255	228	239
2600	3000	0.017	8,000	283	2.62	159	255	235	238
2800	3000	0.017	4,000	260	3.3	78	253	230	244
2400	3270	0.063	15,000	507	2.0	290	249	226	---
2400	3630	0.2	15,000	1,003(c)	2.1	474	260	231	---
2600	3630	0.2	8,000	719(c)	4.17	296	---	---	---
2400	(b)	---	15,000	670	6.03	171	265	227	---
2600	(b)	---	10,000	194	4.2	69.5	263	232	236

NOTE:
(a) Extrapolated value
(b) As-TIG welded, tested with weld bead in longitudinal direction
(c) Test in progress

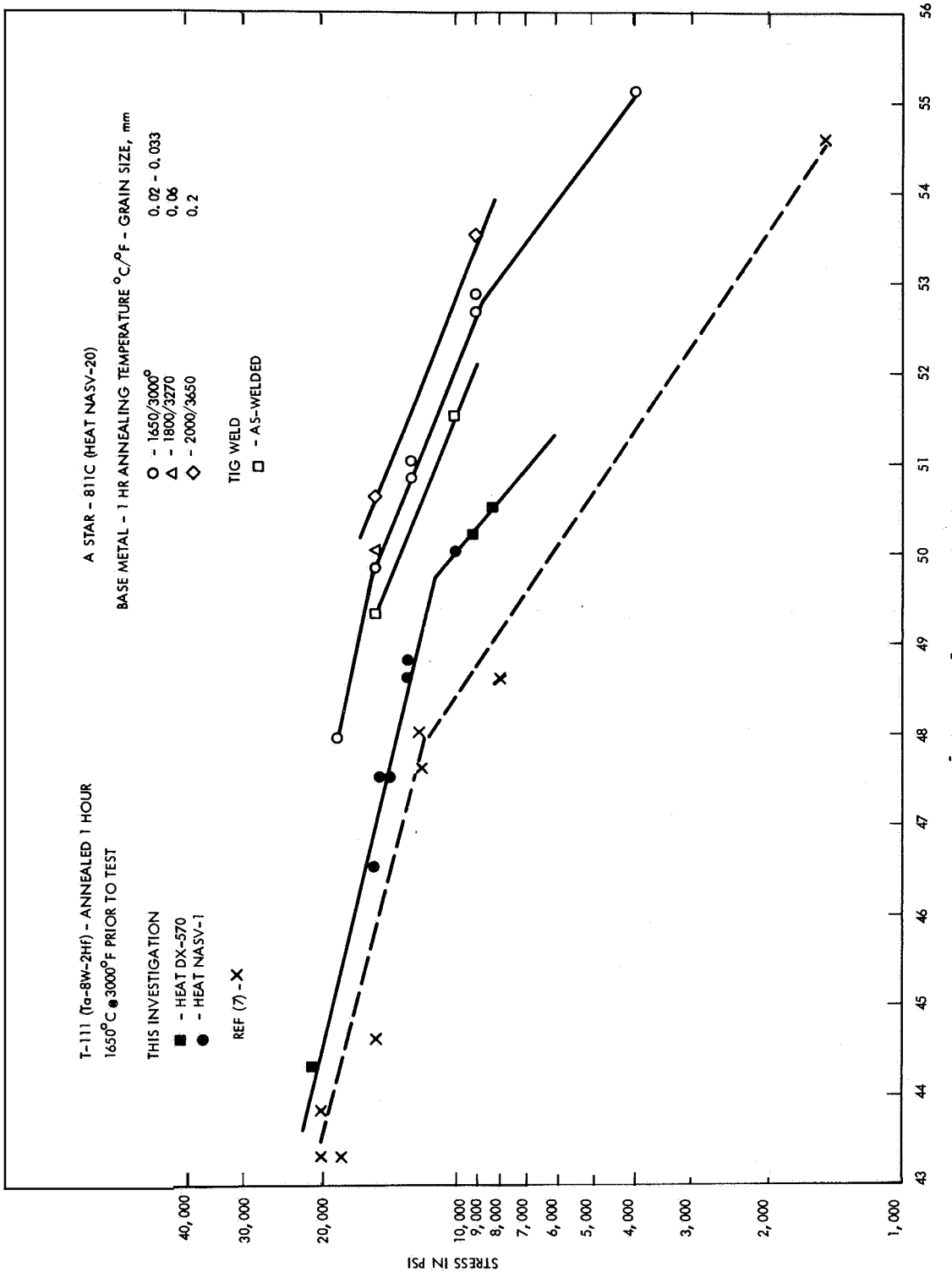


FIGURE 5 - Creep Behavior of Ta-8W-1Re-0.7Hf-0.025C (ASTAR-811C), Heat NASV-20

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Attn: Librarian

Temescal Metallurgical
Berkeley, California
Attn: Librarian

Union Carbide Stellite Corp.
Kokomo, Indiana
Attn: Librarian

Union Carbide Metals
Niagara Falls, New York
Attn: Librarian

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

United Nuclear Corporation
Research & Engineering Center
Grassland Road
Elmsford, New York 10523
Attn: Librarian
Attn: Mr. Albert Weinstein

Astronuclear laboratory

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

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

University of California
Lawrence Radiation Lab.
P. O. Box 808
Livermore, California
Attn: Librarian

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

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

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

Wyman-Gordon Co.
North Grafton, Massachusetts
Attn: Librarian

Wah Chang Corporation
Albany, Oregon
Attn: Librarian

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