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DEVELOPMENT OF DISPERSION STRENGTHENED TANTALUM BASE ALLOY

Thirteenth 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

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THIRTEENTH QUARTERLY PROGRESS REPORT

Covering the Period

November 20, 1966 - February 20, 1967

Prepared For

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ABSTRACT

Development of dispersion strengthened tantalum base alloys for use in advanced space power systems continued as the evaluation of Ta-8W-1Re-1Hf (ASTAR-811) and Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN) sheet material was essentially completed. Tensile properties of TIG welded sheet specimens of both alloys were determined over the temperature range of -320 to 2600°F. One hour post weld anneals at temperatures ranging from 1800 to 2600°F resulted in an increase in the ductile-brittle transition temperature of TIG welded ASTAR-811CN, while the transition temperature of TIG welded ASTAR-811 remained below -320°F. Phase identification studies on ASTAR-811CN indicated that the HCP tantalum dimetal carbide is the precipitate which occurs during processing to 0.04 inch sheet and short time anneals. The FCC carbonitride phase occurs at the expense of the dimetal carbide during longer ($\geq$16 hours) anneals at temperatures of about 2400°F and higher. The scope of work this period was expanded to include an investigation of the effect of grain size and annealing temperatures on the creep properties of ASTAR-811, ASTAR-811CN, and ASTAR-811C (Ta-8W-1Re-0.7Hf-0.025C). Grain size data were obtained on these alloys as a function of annealing time and temperature.

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I. INTRODUCTION

This, the thirteenth quarterly progress report on the NASA-sponsored program, "Development of Dispersion Strengthened Tantalum Base Alloys" describes the work accomplished during the period November 20, 1966 to February 20, 1967. The work was performed under Contract NAS 3-2542.

The primary objective of the current phase of this program is the processing and evaluation of 0.04 inch sheet of three compositions which were melted as 60-pound, 4-inch diameter ingots. The compositions were selected for potential sheet and tubing applications on the basis of weldability, creep resistance, and fabricating characteristics.

Prior to this quarterly period, several promising tantalum alloy compositions were developed which exhibited a good combination of creep resistance, weldability, and fabricability.[1] The three compositions selected for scale up are:

ASTAR-811	Ta-8W-1Re-1Hf
ASTAR-811C	Ta-8W-1Re-1Hf-0.025C
ASTAR-811CN	Ta-7W-1Re-1Hf-0.012C-0.012N

These compositions were consumable electrode double vacuum arc melted as 60-pound, 4-inch diameter ingots, which were subsequently processed to 0.04-inch sheet by a combination of forging and rolling. Evaluation of composition Ta-8W-1Re-1Hf-0.025C (ASTAR-811C) has been essentially completed,[2] and evaluation of the remaining two compositions Ta-8W-1Re-1Hf (ASTAR-811) and Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN), initiated.[3]

During this quarterly period the evaluation of weldability, tensile properties, and creep resistance of the Ta-8W-1Re-1Hf (ASTAR-811) and Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN) was essentially completed. The scope of the contract was also expanded to include an investigation of the effect of grain size on the creep properties of all three scale-up alloys. To date grain size data has been obtained as a function of annealing time and temperature.

II. PROGRAM STATUS

A. EFFECT OF THERMAL TREATMENT ON CREEP BEHAVIOR

The scope of work was changed to include a limited investigation on the effect of final annealing treatment on the creep behavior of the ASTAR-811, ASTAR-811C, and ASTAR-811CN compositions. Prior work[4] on ASTAR-811C (Ta-8W-1Re-1Hf-0.025C) has shown that creep behavior is strongly influenced by the final annealing temperature. Increasing the final annealing temperature from 1650°C to 2000°C resulted in a 50% reduction in creep rate. However, the average grain diameter increased from 0.03 mm to 0.18 mm as the 1 hour annealing temperature was increased from 1650°C to 2000°C. In addition there was also a significant change in the precipitate morphology as the annealing temperature was increased.[4] Thus it is important to identify the factors contributing to the observed improvement in creep behavior. A series of specimens will be annealed over the temperature range of 1800-2100°C for a time sufficient to produce a final grain size of 0.03 mm, the resulting grain size achieved after the standard final annealing treatment, i.e., 1 hour at 1650°C. The specimens will then be creep tested at 2400°F under an applied stress of 15,000 psi.

During this period, 0.04-inch thick sheet specimens of all three ASTAR compositions were annealed at 1800, 1900, 2000, and 2100°C (3270, 3450, 3630, and 3810°F) for 30, 300, and 900 seconds. The 0.04-inch sheet from which the specimens were taken had been reduced 85% by cold rolling. The average grain diameter determined by the line intercept method and room temperature hardness data are recorded in Table 1. The annealing sequence consisted of slowly heating the furnace to 1200°C while maintaining the chamber pressure at $\leq 1 \times 10^{-5}$ torr and then heating to the desired temperature as rapidly as possible. Time at temperature was recorded from when the specimen reached a temperature within 50°C of the test temperature. After the specified time at temperature the furnace was shut off and the chamber was backfilled with helium gas to accelerate cooling of the specimens. All specimens were heat treated bare. From these results the heat treatments selected to produce a grain size of 0.03 mm are

TABLE 1 - Effect of Annealing Temperature and Annealing Time on the Grain Size of ASTAR-811, ASTAR-811C, and ASTAR-811CN

Composition	Annealing Time (min.)	Grain Size (in mm) and Hardness[1] for Temperatures, °C/°F							
		1800		1900		2000		2100	
		GS	DPH	GS	DPH	GS	DPH	GS	DPH
ASTAR-811 (Ta-8W-1Re-1Hf)	1/2	0.016	196	0.024	199	0.029	202	0.042	199
	5	0.024	200	0.038	196	0.077	197	0.098	195
	15	0.045	181	0.079	194	0.118	182	0.186	197
ASTAR-811C (Ta-8W-1Re-0.7Hf-0.025C)	1/2	0.016	263	0.021	262	0.033	270	0.038	254
	5	0.026	267	0.030	258	0.067	268	0.093	256
	15	0.045	271	0.065	251	0.100	281	0.178	255
ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N)	1/2	0.013	287	--	--	0.031	305	--	--
	5	0.026	294	--	--	0.067	288	--	--
	15	0.039	297	--	--	0.107	272	--	--

(1) DPH, 30 Kg load.

as follows:

10 minutes at 1800°C/3270°F
5 minutes at 1900°C/3450°F
30 seconds at 2000°C/3630°F

It has been shown that under isothermal conditions, the grain size (D) varies with time (t) according to the following expression:

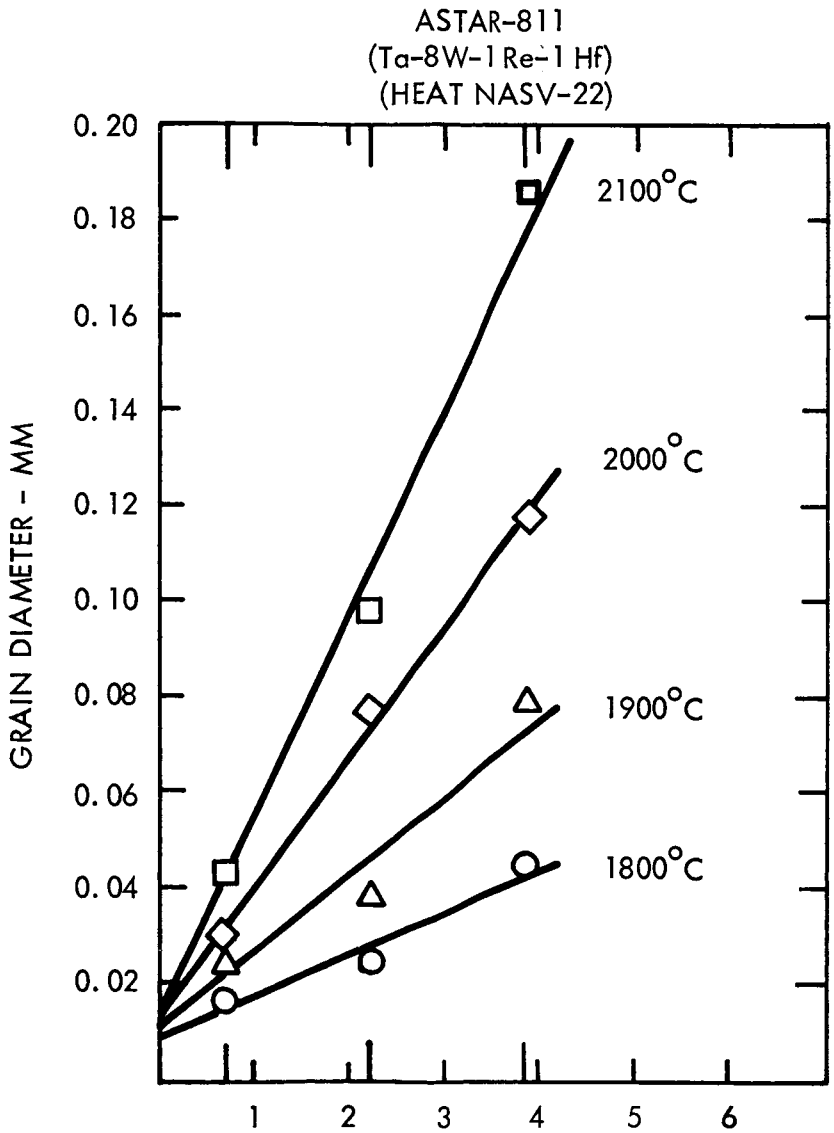
$$D^2 = K \gamma V t$$

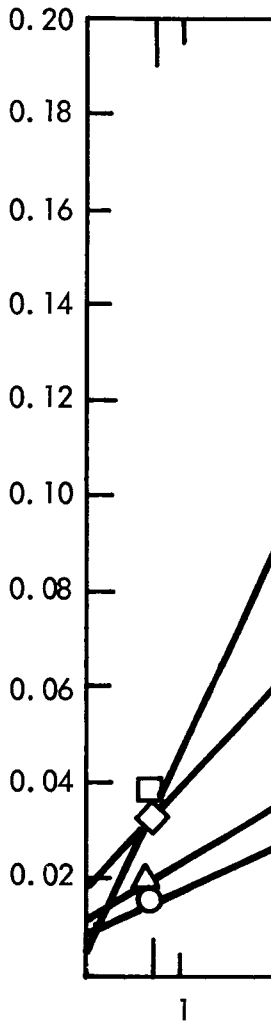
where the surface energy of the boundary (γ) is the driving force for boundary migration. K is a rate constant and V is the grain atomic volume. Thus at a given temperature, the grain size is proportional to the square root of time. The grain size data in Table 1 when plotted as a function of time (see Figure 1) result in a good linear fit. The slope of this curve $\frac{\partial D}{\partial t}^{1/2}$ is equal to $\sqrt{K}$, the parabolic rate constant which varies with temperature according to the familiar Arrhenius rate equation;

$$K = K_o \exp^{-Q/RT}$$

where Q is the activation energy, T is absolute temperature, R is the gas constant, and K_o a proportionality constant. Thus the activation energy for grain growth can be calculated from the slope of the curve D^2/t vs 1/T. From the D^2/t vs 1/T plot in Figure 2, a value of Q of 92 kcal/mole was determined. The activation energy for the self diffusion of tantalum is reported to be 110 kcal/mole[5] and it would be expected that the activation energy for grain boundary migration would be less than one-half this value. Thus the value of 92 kcal/mole determined for these tantalum alloys appears to be higher than would normally be expected. The explanation for this apparent high value is not evident.

The grain growth behavior of all three tantalum compositions was identical over the range of test temperatures studied. This would be expected for the ASTAR-811 and ASTAR-811CN since at 1800°C, the solvus for the 0.012C and 0.012N has been exceeded. However, the carbon solvus for the ASTAR-811C which contains 0.025% is not exceeded until heating


ASTAR-811
(Ta-8W-1 Re-1 Hf)
(HEAT NASV-22)
GRAIN DIAMETER - MM
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
1
2
3
4
5
6
2100°C
2000°C
1900°C
1800°C


(Ta-
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
1

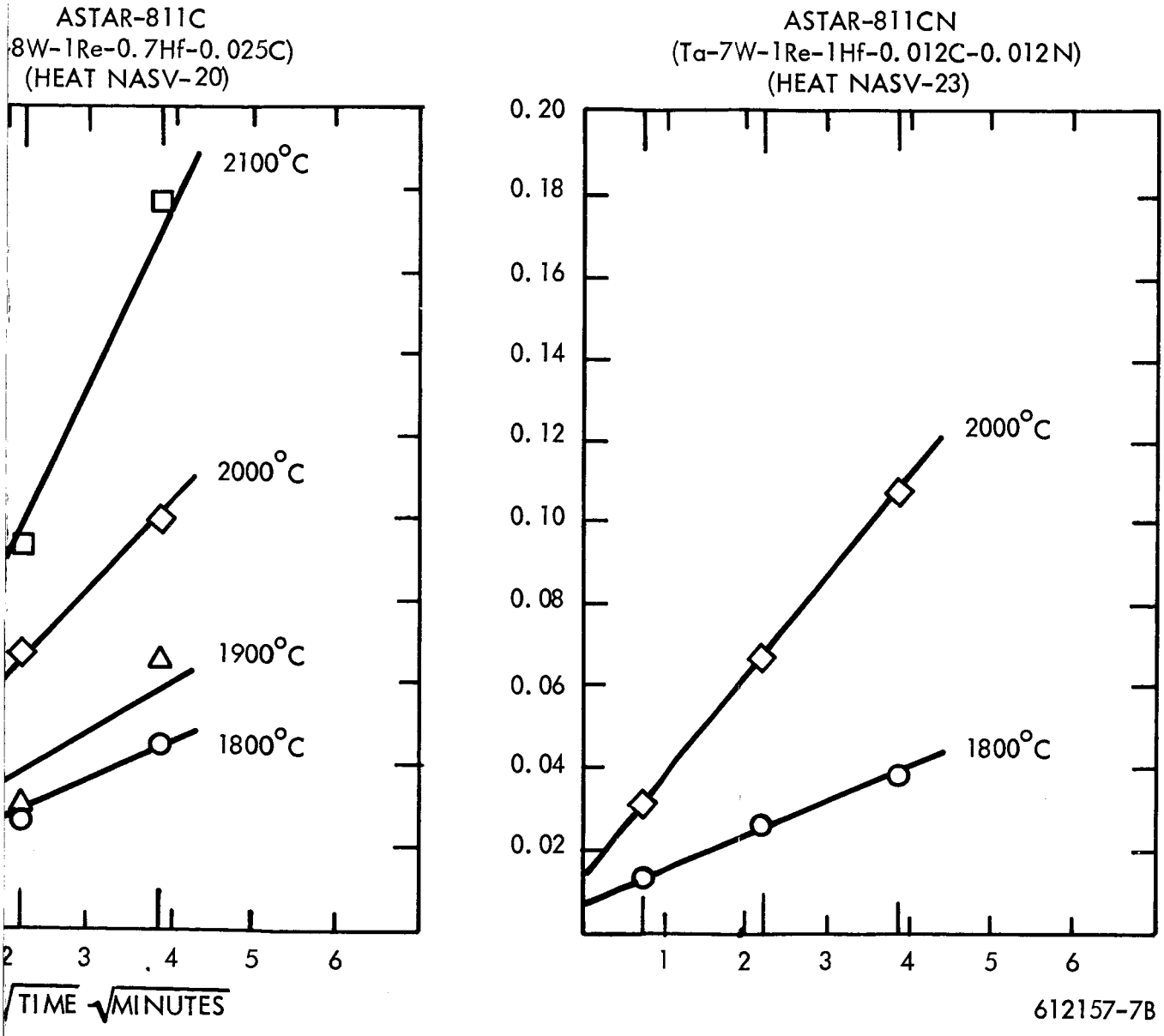


FIGURE 1 - Grain Size of ASTAR-811, ASTAR-811C, and ASTAR-811CN as a Function of Annealing Time and Temperature

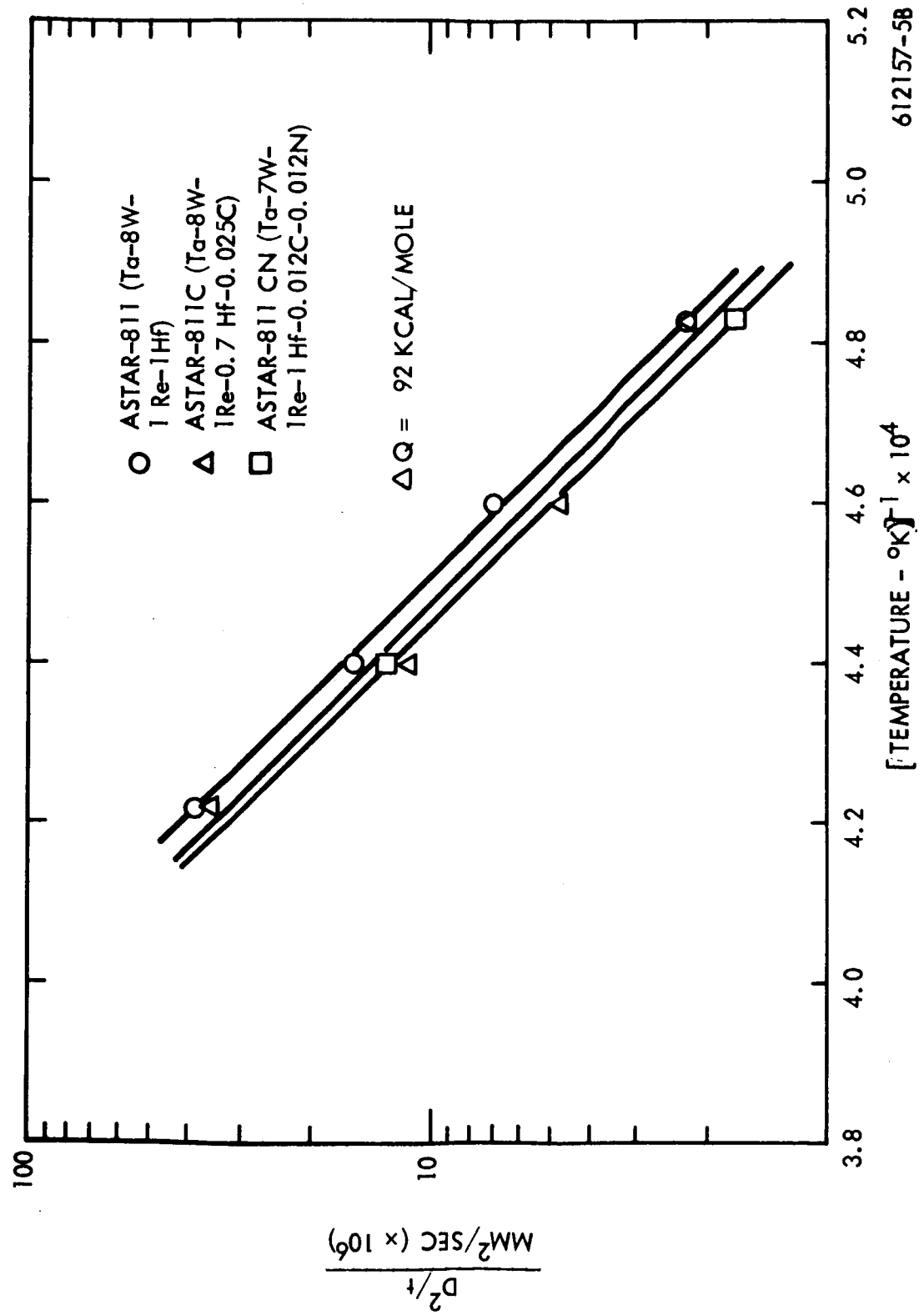


FIGURE 2 - Parabolic Rate Constant for Grain Growth of ASTAR-811, ASTAR-811C, and ASTAR-811CN as a Function of Reciprocal Temperature

above 1900°C. Thus it can be concluded that the carbide particles present at 1800°C do not inhibit grain boundary migration.

B. INTERSTITIAL ELEMENT LOSSES DURING VACUUM HEAT TREATMENT

The carbide and nitride phases in the tantalum alloy matrix have appeared stable for long time periods over the temperature range of 1800-2600°F when exposed at pressures of $\leq 1 \times 10^{-8}$ torr.[6] However, as discussed previously, the optimum creep characteristics are achieved by using final annealing treatments in the 3200-3600°F temperature range. Current industry wide practice for high temperature vacuum heat treatment is to use unbaked, polymer sealed systems operating at 10^{-5} - 10^{-4} torr. Most heat treatment specifications also require Ta, Cb, or Cb-1Zr foil wrapping of the work piece as a barrier to contamination during the annealing cycle. Decarburization of a carbon containing tantalum alloy at 1×10^{-5} torr at >2000°C was reported early in this investigation.[7] It is assumed that the decarburization is via the methane and/or CO reactions similar to that reported for decarburization of molybdenum alloys. It was also shown that wrapping the sample with pure tantalum foil resulted in a greater carbon loss. It is assumed that the foil acts as a sink and that the rate of carbon transfer across the foil interface is faster than the carbon loss due to reaction with the residual H_2, H_2O present in the vacuum chamber atmosphere at 1×10^{-5} torr.

Additional vacuum annealing tests were made during the report period on the carbonitride strengthened composition ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N). Sheet specimens, 0.04-inch thick, bare and wrapped with tantalum foil, were exposed for 5, 30, and 60 minutes at 2100°C (3810°F). After heat treatment, the samples were then analyzed for carbon and nitrogen content. The analytical results are recorded in Table 2 and graphically illustrated in Figures 3 and 4. Significant carbon and nitrogen losses have occurred during this annealing treatment and confirm the losses observed previously when annealing this composition at 2000°C and above.[3] The use of a foil wrapping, while reducing the possibility of oxygen contamination, acts as a sink for carbon. The nitrogen loss is assumed to be by degassing and the foil wrapping appears to retard the rate at which it occurs.

TABLE 2 - Chemical Analyses of Unwrapped and Wrapped[a] ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) as a Function of Time at 2100°C/3810°F and 1×10^{-5} Torr in Oil Diffusion Pumped Vacuum System

Condition[b]	Time (min.)	Carbon Content (ppm)	% Carbon Lost	Nitrogen Content (ppm)	% Nitrogen Lost	Total Interstitials Lost	
						ppm	(%)
Unwrapped	5	96	20	64	47	80	33.5
	30	64	47	49	59	127	53
	60	55	54	31	74	154	64
Wrapped[a]	5	72	40	113	6	55	23
	30	82	32	70	42	88	37
	60	47	61	64	47	129	54

(a) Specimens wrapped tightly in pure tantalum foil.
(b) Initial carbon and nitrogen content 120 ppm.

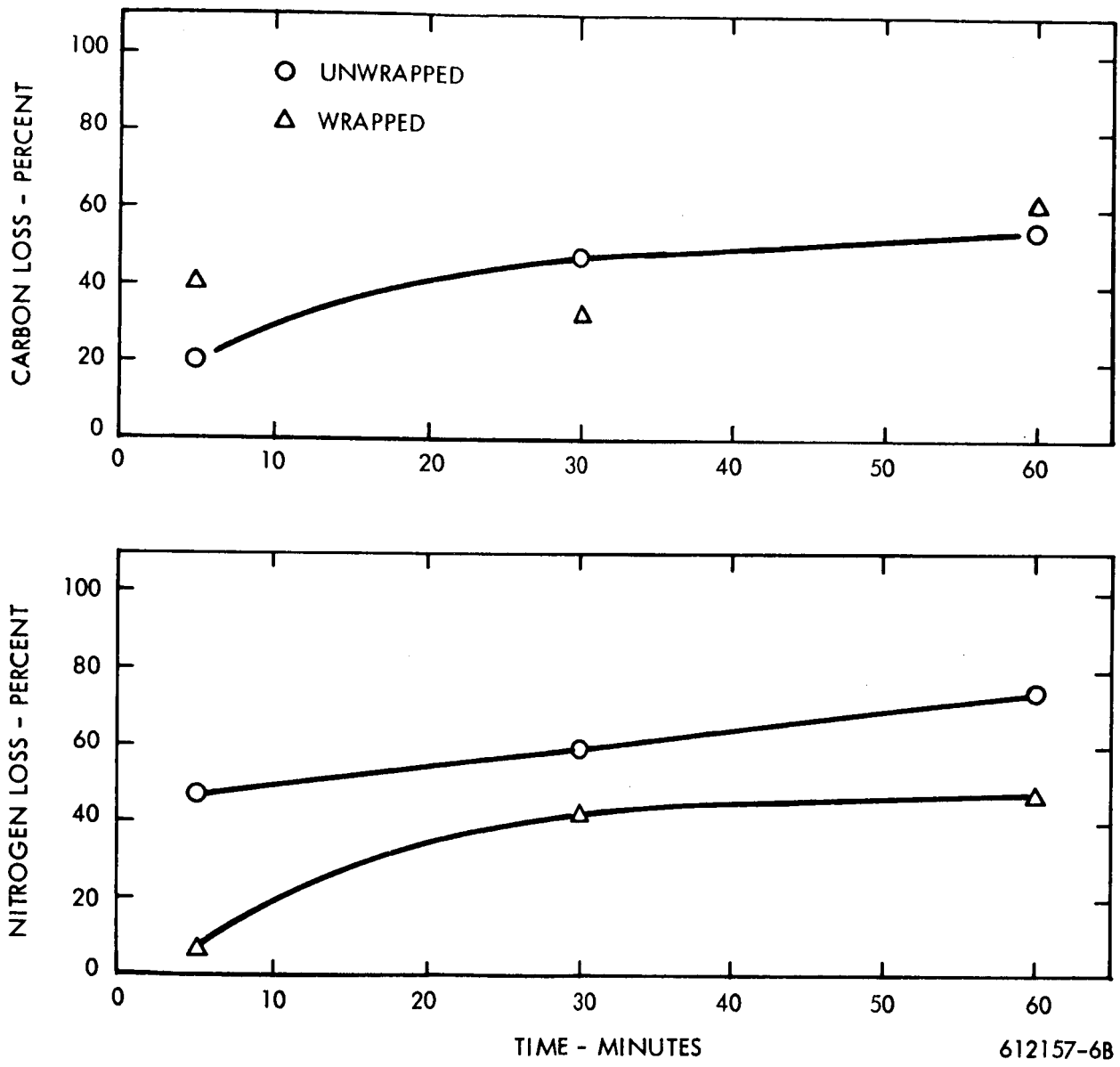


FIGURE 3 - Carbon and Nitrogen Losses in Unwrapped and Wrapped ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) as a Function of Time at 2100°C/3810°F and 1 x 10^{-5} Torr in Oil Diffusion Pumped System

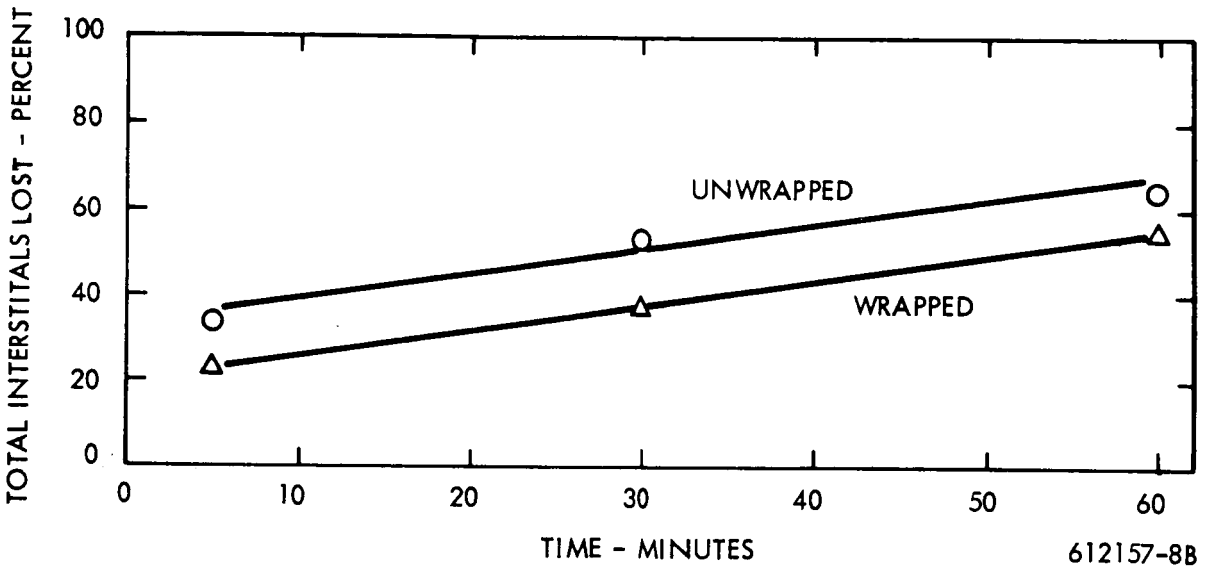


FIGURE 4 - Total Interstitial Losses in Unwrapped and Wrapped ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) as a Function of Time at 2100°C/3810°F and 1 x 10^{-5} Torr in Oil Diffusion Pumped System

These tests are very preliminary in nature and more detailed work is beyond the scope of this program. But certain obvious implications arise if refractory metal alloys strengthened with carbide and/or nitride dispersions are to be utilized to the fullest. High temperature vacuum annealing treatments on alloys of the type which have been developed during this investigation may have to be accomplished in bakeable ultra-high vacuum systems to prevent carbon loss and with a controlled nitrogen partial pressure to prevent nitrogen loss. There is no doubt that a general improvement in the current state-of-the-art of vacuum annealing as practiced by the industry will have to be advanced in order to ensure that interstitial composition does not change during the required annealing treatment. This advancement will necessarily include the development of large bakeable ultra high vacuum furnaces capable of operation at up to approximately 4000°F.

C. WELDABILITY

The effect of post weld annealing on the ductile-brittle transition temperature was determined on 0.04-inch sheet of ASTAR-811 and ASTAR-811CN which had been annealed for 1 hour at 1650°C and then TIG welded. Bead-on-plate type welds with 100% penetration were tested in bending over a 1t bend radius with the weld bead transverse to the bend axis. After welding specimens were annealed for 1 hour at 980, 1200, and 1425°C (1800, 2200, and 2600°F). The data obtained are recorded in Table 3. The ductile-brittle transition temperature of the solid solution alloy ASTAR-811 was less than -320°F as-TIG welded and did not change as a result of post-weld annealing. However, the transition temperature of the as-TIG welded ASTAR-811CN increased significantly after post-weld annealing with the higher post weld annealing temperature resulting in the greatest change. The increase in the ductile-brittle transition temperature for ASTAR-811CN summarized below is illustrated in Figure 5.

DBTT (°F)	1 Hour Post Weld Annealing Temperature (°F)
-225	As-Welded
-200	1800
-100	2200
< 0	2600

TABLE 3 - Ductile-Brittle Transition Temperature of Post (TIG) Weld Annealed NASV-23[(a)]
(Ta-7W-1Re-1Hf-0.012C-0.012N)

Post Weld Anneal	Test Temperature (°F)	Test Temperature (°C)	No Load Bend Angle (Degrees)	Remarks	DBTT (°F)	DBTT (°C)
As-TIG Welded	-250	-157	90	Failure		
	-225	-143	91	Bend	-225	-143
1 Hr. at 980°C 1800°F	-100	-73	92	Bend		
	-200	-129	92	Bend	-200	-129
	-225	-143	77	Brittle failure in weld		
	-250	-157	44	Brittle failure in weld and HAZ		
	-320	-196	30	Brittle failure in weld and HAZ		
1 Hr. at 1200°C 2200°F	-100	-73	91	Bend	-100	-73
	-125	-87	77	Brittle failure in weld		
	-150	-101	26	Brittle failure in weld and HAZ		
	-200	-129	37	Brittle failure in weld and HAZ		
1 Hr. at 1425°C 2600°F	75	23	92	Bend		
	50	10	90	Very slight ductile failure in weld		
	25	-4	90	Very slight ductile failure in weld		
	0	-18	90	Very slight ductile failure in weld	<0	<-18
	-100	-73	52	Brittle failure in weld and HAZ		

(a) Specimens were all annealed for 1 hour at 1650°C (3000°F) prior to welding.

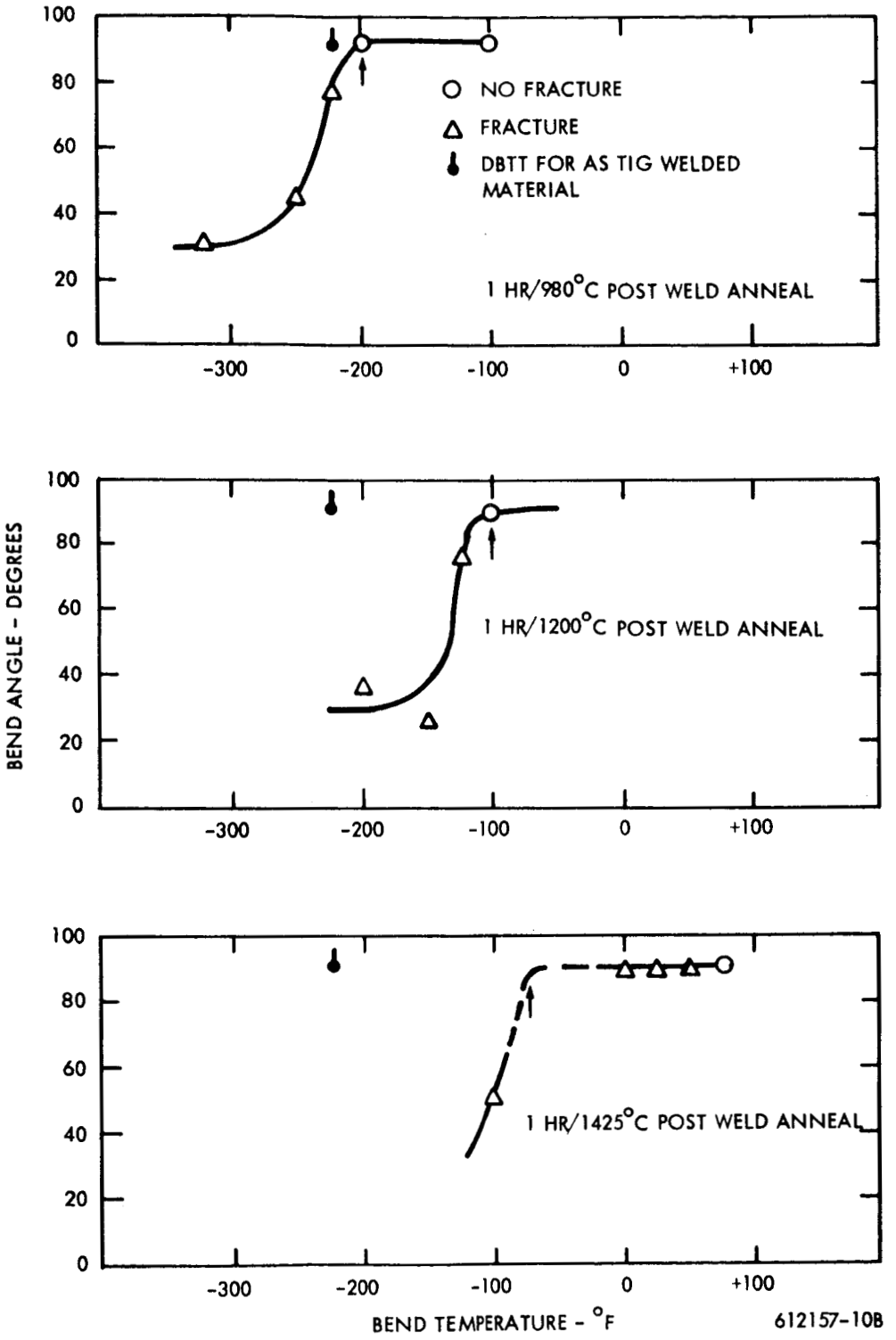


FIGURE 5 - Ductile-Brittle Transition Temperature Test Results for Post (TIG) Weld Annealed ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N)

Failure occurred primarily within the weld and heat affected zones and appeared to be intergranular in nature.

Hardness traverses were made on transverse sections of as-electron beam and as-TIG welded sheet and post-weld annealed specimens of both alloys. The hardness traverses for as-electron beam and as-TIG welded ASTAR-811 and ASTAR-811CN sheet are shown in Figure 6. Little hardness variation was observed in either alloy across the base metal, weld, and heat affected zone. This same behavior was exhibited by the specimens which were post-weld annealed, although there was a decrease in hardness level of from 275 to 255 DPH for the ASTAR-811CN while that of the ASTAR-811 remained essentially unchanged.

The microstructures of the as-TIG welded ASTAR-811 and ASTAR-811CN were essentially single phase in the base metal, fusion, and heat affected zones. The few isolated precipitates observed are assumed to be primarily HfO_2 in the ASTAR-811 and Ta_2C in the ASTAR-811CN. The photomicrographs in Figure 7 are typical of microstructures which were observed for ASTAR-811 and ASTAR-811CN in the as-welded condition.

The microstructure of the as-TIG welded ASTAR-811 remained essentially unchanged after the post weld annealing treatment. An exception however was that a sub-boundary network formed in the heat affected zone and some precipitation occurred near the weld/heat affected zone interface after post weld annealing for 1 hour at 2200°F (see Figure 8). Thus the post weld annealing treatments did not alter the ductile-brittle transition temperature of ASTAR-811 which is consistent with the observed absence of hardness and microstructural changes.

Post weld annealing ASTAR-811CN however did produce significant changes in the as-TIG welded single phase microstructure. The resulting microstructures produced by the 1 hour post weld anneals at 1800, 2200, and 2600°F are shown in Figure 9. Extensive precipitation occurred throughout the base metal, fusion, and heat affected zones during each of the post

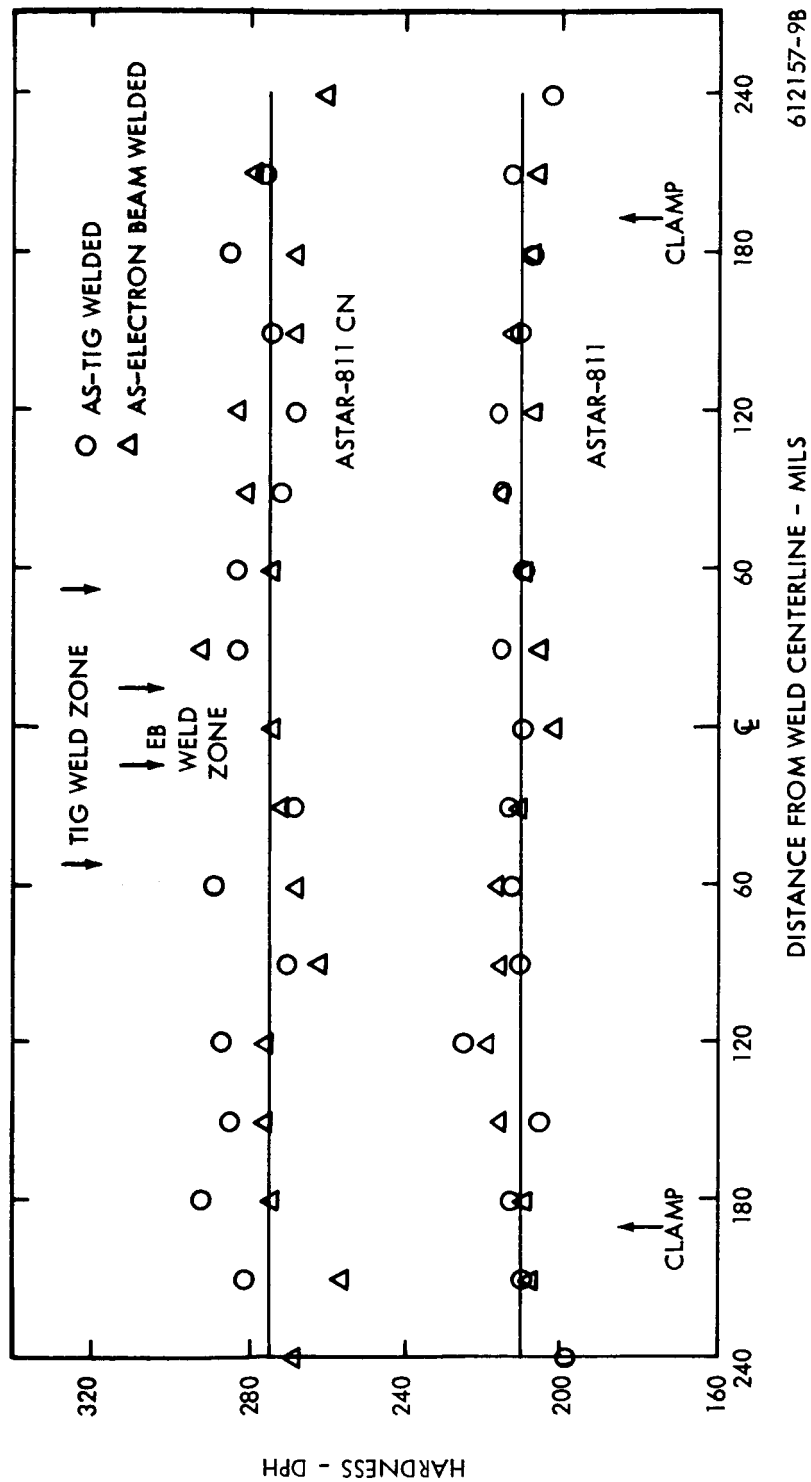
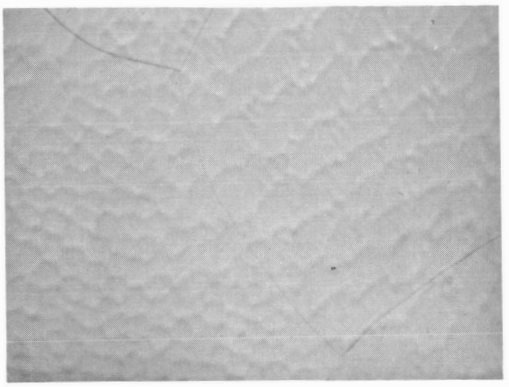
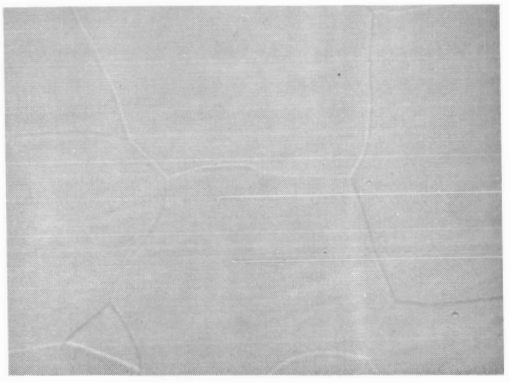


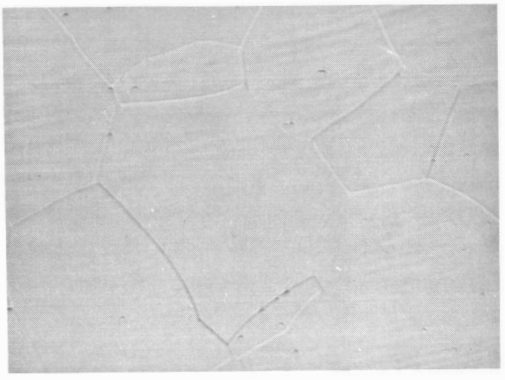
FIGURE 6 - Results of Hardness Traverses Across As-Welded Specimens of ASTAR-811 (Ta-8W-1Re-1Hf) and ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N)



(a) Weld Zone



(b) Heat Affected Zone

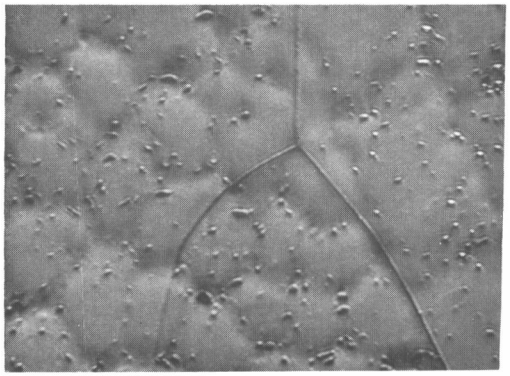


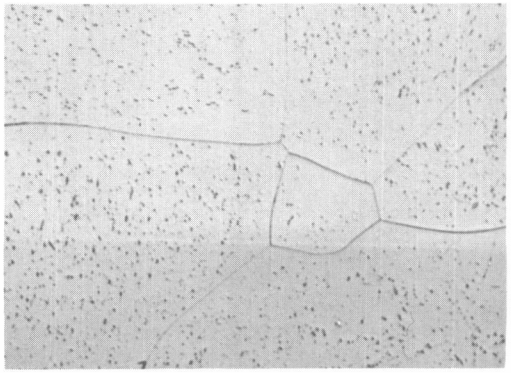
(c) Base Metal

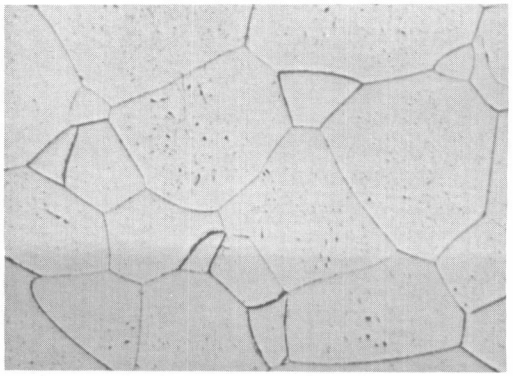
FIGURE 7 - Representative Microstructures of TIG Welded ASTAR-811 (Ta-8W-1Re-1Hf) 500X

1500X

FIGURE 8 - Microstructure of Weld Zone/Heat Affected Zone Interface in TIG Welded ASTAR-811 (Ta-8W-1Re-1Hf) Specimen After 1 Hr. Post Weld Anneal at 1200°C (2200°F)

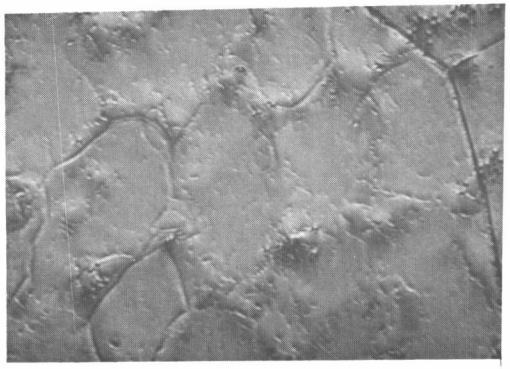


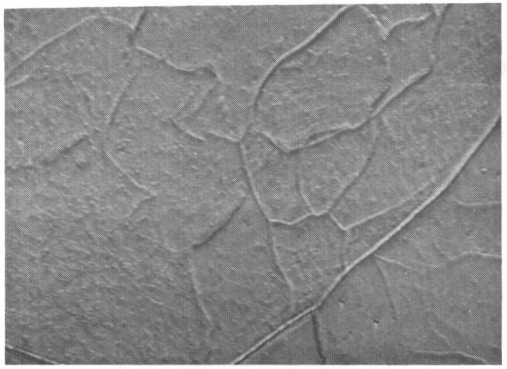


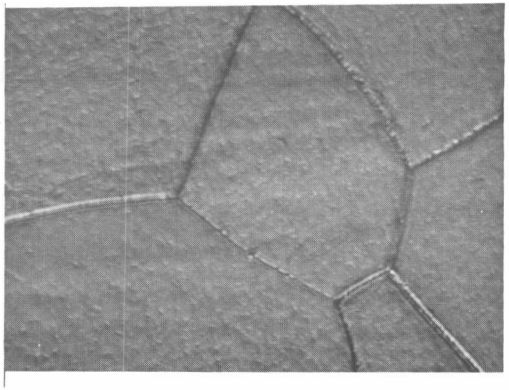


Weld Zone 1500X HAZ 500X Base Metal 500X

Post Weld Annealed for 1 Hour at 980°C (1800°F)



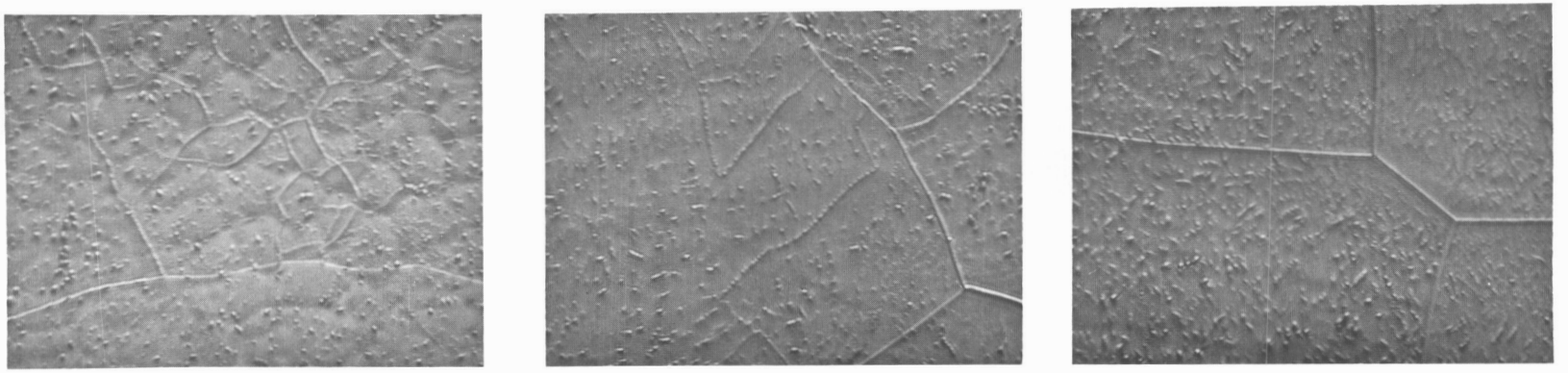




Weld Zone 1500X HAZ 1500X Base Metal 1500X

Post Weld Annealed for 1 Hour at 1200°C (2200°F)

FIGURE 9 - Microstructures of TIG Welded ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Specimens After 1 Hr. Post Weld Anneals at Various Temperatures



Weld Zone 1500X HAZ 1500X Base Metal 1500X

Post Weld Annealed for 1 Hour at 1425°C(2600°F)

FIGURE 9 (continued) - Microstructures of TIG Welded ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Specimens After 1 Hr. Post Weld Anneals at Various Temperatures

weld annealing treatments. The formation of this precipitate, which is most likely Ta_2C, no doubt accounts for the observed hardness decrease.

The distribution of the precipitate however did appear to be affected by the annealing temperature. At 1800°F, the precipitation occurred primarily throughout the matrix while at 2200°F and 2600°F, precipitates were formed at primary grain boundaries and cell and sub-boundaries in the fusion and heat affected zones. The cell and sub-boundary precipitates were larger after annealing at 2600°F. This change in precipitate distribution can thus be used to qualitatively explain the increase in ductile-brittle transition temperature with increasing post weld annealing temperature, particularly as the annealing temperature was increased from 1800 to 2200°F.

D. MECHANICAL PROPERTIES

1. Tensile Properties of TIG Welds — Bead-on-plate tungsten inert gas (TIG) welded specimens of ASTAR-811 and ASTAR-811CN were tested over the temperature range of -320 to 2600°F (-195 to 1425°C) to evaluate the effects of welding on tensile properties. The welds, both longitudinal and transverse, were made on 0.04-inch thick sheet which had been annealed for 1 hour at 1650°C (3000°F) prior to welding. Tensile data are recorded in Table 4A along with the previously obtained data for ASTAR-811C. The tensile data for both ASTAR-811 and ASTAR-811CN appear to be anomalous in that the ductility values at room temperature were lower than those obtained at -195°C (-320°F).

An additional set of room temperature tungsten inert gas (TIG) welded tensile specimens of each alloy were made and retested because of these anomalous results. Sections of 0.33-inch plate, processed from the side forgings, were annealed for 1 hour at 1700°C (3090°F) and rolled to 0.05-inch sheet, from which specimen blanks were obtained. These blanks were then annealed for 1 hour at 1650°C (3000°F) and the required transverse and longitudinal bead-on-plate TIG welds were made. Specimens, 0.04-inch thick, were machined from the welded blanks, x-rayed to ensure that no weld defects were present, and tested at room

TABLE 4A - Tensile Properties of TIG Welded ASTAR-811, ASTAR-811C, and ASTAR-811CN

Composition and Heat No.	Test Temp. (°F)	Weld Direction	0.2% Yield Strength (ksi)	Ultimate Tensile Strength (ksi)	% Elongation	
					Uniform	Total
ASTAR-811 (Ta-8W-1Re-1Hf) Heat NASV-22	-320	Longitudinal	133.7	157.0	16.3	18.6
	-320	Transverse	137.3	160.6	16.0	19.7
	RT	Longitudinal	77.3	---	8.15	8.3
	RT	Transverse	80.5	---	8.3	8.6
	1800	Longitudinal	36.2	57.6	---	18.1
	2000	Transverse	30.7	47.2	---	10.7
	2400	Longitudinal	27.8	32.2	---	6.6
	2600	Transverse	24.3	28.5	---	4.9
ASTAR-811C (Ta-8W-1Re-0.7Hf-0.025C) Heat NASV-20	-320	Longitudinal	157.3	184.6	16.7	24.2
	-320	Transverse	159.0	176.2	10.9	14.2
	RT	Longitudinal	109.8	115.3	15.0	28.5
	RT	Transverse	89.3	107.2	10.6	18.7
	1800	Longitudinal	44.0	67.1	---	18.7
	2400	Longitudinal	35.3	41.1	---	29.0
	2600	Longitudinal	32.5	36.0	---	26.7
ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Heat NASV-23	-320	Longitudinal	166.4	---	5.25	5.4
	-320	Longitudinal	163.0	194.5	17.4	22.5
	RT	Transverse	106.3	113.5	9.7	11.3
	RT	Transverse	110.8	116.3	9.6	15.4
	1800	Longitudinal	45.3	70.6	---	20.6
	2000	Transverse	40.0	65.0	---	16.4
	2400	Longitudinal	37.2	45.4	---	19.7
	2600	Transverse	31.0	35.9	---	21.5

TABLE 4B - Tensile Properties of TIG Welded ASTAR-811 and ASTAR-811CN

Composition and Heat No.	Test Temperature (°F)	Weld Direction	0.2% Yield Strength (ksi)	Ultimate Tensile Strength (ksi)	% Elongation	
					Uniform	Total
ASTAR-811 (Ta-8W-1Re-1Hf) Heat NASV-22	-320	Longitudinal	134.6	152.3	17.50	22.15
	-320	Transverse	137.0	148.0	11.25	14.35
	RT	Longitudinal	77.6	92.3	14.60	19.15
	RT	Transverse	79.2	90.9	9.20	11.95
ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Heat NASV-23	-320	Longitudinal*	162.0	188.0	4.85	5.50
	-320	Transverse*	158.4	189.8	3.70	5.90
	RT	Longitudinal	109.0	116.0	13.15	22.70
	RT	Transverse	110.0	114.0	3.65	5.95

*The stress-strain curves indicated that twinning may have occurred.

temperature and at -320°F. The results are recorded in Table 4B. Where significant differences were noted, they are assumed to represent defective weldments in the original specimens. The test results for the ASTAR-811CN weldments indicate that the fracture mode is changing from ductile to brittle at -320°F.

2. Creep Properties — Creep properties for 0.04-inch thick ASTAR-811 and ASTAR-811CN sheet were obtained at 2200-2600°F at stress levels of 8,000-19,000 psi. The data are recorded in Table 5. These data normalized using the Larson-Miller parameter are plotted in Figure 10 along with data for T-111 and ASTAR-811C. The creep strength of the solid solution composition ASTAR-811 is slightly better than T-111 but significantly inferior to both ASTAR-811C and ASTAR-811CN. The effects of the individual additions on creep strength at 2400°F and 15,000 psi are summarized below.

Composition w/o	Time to 1% Strain at 2400°F and 15,000 psi
Ta-8W-2Hf (T-111)	20
Ta-8W-1Re-1Hf (ASTAR-811)	54
Ta-8W-1Re-1Hf-0.025C (ASTAR-811C)	260
Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN)	157

Although there is an apparent increase in creep resistance when rhenium is added to the tantalum matrix the increase may be due to the reduction of hafnium content, which has already been shown to exert a pronounced affect on creep strength.[1] There is however no doubt concerning the effect of the carbon and/or nitrogen additions on creep strength. Additional tests are underway to establish the stress and temperature dependence for each composition.

E. PHASE IDENTIFICATION

Information as to the identity of the precipitating phase(s) in the tantalum alloy matrix and their stability as a function of temperature and time is of vital importance.

TABLE 5 - Creep Properties of ASTAR-811 and ASTAR-811CN[(a)]

Composition	Test Temperature (°F)	Stress (psi)	Test Duration (hrs.)	Total Elongation (%)	Time to 1% Strain (hrs.)	Vickers Hardness DPH	
						Pre-Test	Post Test
ASTAR-811 (Ta-8W-1Re-1Hf) (Heat NASV-22)	2200	19,000	826	6.4	185	213	212
	2200	15,000	1670	0.66	---	213	209
	2400	15,000	165	4.6	54		
	2400	12,766	377	1.3	317	209	213
ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) (Heat NASV-23)	2200	19,000	835	2.9	607	294	241
	2400	15,000	426	7.3	157	287	228
	2400	12,500	530	1.43	403	296	228
	2600	8,000	382	5.4	172	290	245

(a) Specimens annealed at 1650°C for 1 hr. at 1×10^{-5} torr then tested at $<1 \times 10^{-8}$ torr.

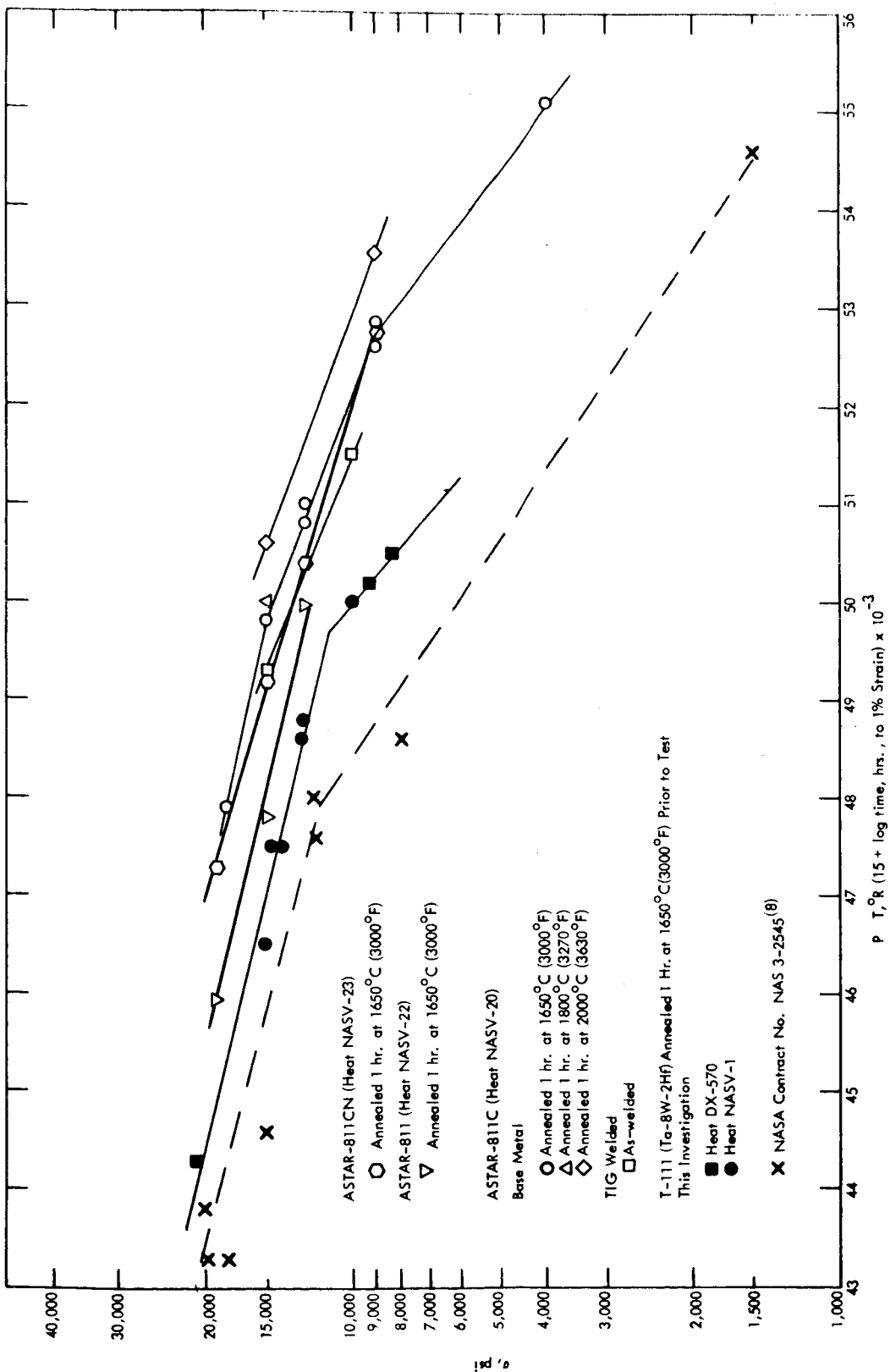


FIGURE 10 - Creep Properties of ASTAR Tantalum Alloys

Understanding of these phase relationships will aid in identifying the mechanism(s) contributing to the low and elevated temperature strength thus allowing more precise control over the final mechanical properties.

Prior work on the Ta-8W-1Re-1Hf-0.025C (ASTAR-811C) composition has shown that the dimetal carbide (Ta_2C) is the only precipitating phase. It was also shown that the morphology of the Ta_2C precipitate could be significantly altered by thermal treatment.[4]

During this period, phase identification work has been carried out on the Ta-7W-1Re-1Hf-0.012C-0.012N composition (ASTAR-811CN). This alloy is essentially identical to ASTAR-811C except one half of the carbon has been replaced with an equivalent amount of nitrogen.

The dispersed phases present in the ASTAR-811CN at the various stages of processing from the as-cast ingot to 0.04 inch sheet were chemically extracted and analyzed by x-ray diffraction. The results which are recorded in Table 6 indicate only the presence of the HCP dimetal carbide Ta_2C (a_o = 3.10 - 3.11 Å, c_o = 4.94 Å, c/a = 1.59). There is however very minor amounts of monoclinic HfO_2 present which no doubt results from the residual oxygen. The amount of Ta_2C which occurred after annealing sheet at 3000°F and above is generally quite small since the carbon solvus for ASTAR-811CN is exceeded at or slightly above this temperature. No evidence of a nitride phase was detected and is consistent with previous work on tantalum alloy compositions containing 100-150 ppm nitrogen.[9]

Subsequent aging at lower temperatures was carried out over the temperature range of 2000-2600°F for times of 1 and 16 hours on ASTAR-811CN 0.04 inch sheet which had been solution annealed at 3000°F for 1 hour. Longer time aging treatments (up to 1000 hours) over this same temperature range are in progress and will be completed during the next report period. X-ray diffraction results on the residues chemically extracted from these specimens are listed in Table 7. Only Ta_2C was observed after aging for 1 and 16 hours up to 2400°F

TABLE 6 - X-ray Diffraction Analyses of Various ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Bulk Extracted Residues

Specimen	Phases	Comments
As-cast	HCP Ta_2C	1
As-upset forged at 1400°C (2550°F)	HCP Ta_2C Mono HfO_2(VW)	1
Forged + annealed (1 hr./1650°C (3000°F)	HCP Ta_2C Mono HfO_2(VW)	1
As-rolled 0.06" sheet	HCP Ta_2C	1
0.06" sheet annealed (1 hr./1700°C (3090°F)	HCP Ta_2C	1
As-rolled 0.04" sheet	HCP Ta_2C Mono HfO_2(VVW)	1
0.04" sheet annealed (1 hr./1650°C (3000°F)	HCP Ta_2C	1

NOTE: Approximate lattice parameters of all the HCP Ta_2C phases were:

a_o = 3.10 to 3.11 Å Co = 4.94 Å c/a = 1.59

VW - VeryWeak, VVW - Very Very Weak

(1) Diffraction lines partially resolved

TABLE 7 - X-ray Diffraction Analyses of Phases Extracted from ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Specimens Annealed for 1 Hour at 1650°C and Aged for 1 and 16 Hours at 1090, 1200, 1315, and 1425°C.

Specimen	Phases	Comments
0.04" sheet annealed (1 hr./1650°C (3000°F)	HCP Ta_2C	1
+(1 hr./1090°C (2000°F)	HCP Ta_2C	1
+(1 hr./1200°C (2200°F)	HCP Ta_2C	
+(1 hr./1315°C (2400°F)	HCP Ta_2C Mono HfO_2(VVW)	
+(1 hr./1425°C (2600°F)	HCP Ta_2C Mono HfO_2(VW)	1
+(16 hrs./1090°C (2000°F)	HCP Ta_2C	
+(16 hrs./1200°C (2200°F)	HCP Ta_2C Mono HfO_2(VVW)	
+(16 hrs./1315°C (2400°F)	HCP Ta_2C Mono HfO_2(VVW)	1
+(16 hrs./1425°C (2600°F)	HCP Ta_2C (S) Mono HfO_2(VW) FCC Hf(CN) (M)	1 a_o=4.56 Å

NOTE: Approximate lattice parameters of all the HCP Ta_2C phases were:

a_o=3.10 to 3.11 Å Co =4.94 Å c/a =1.59

(1) Diffraction lines partially resolved

and after 1 hour at 2600°F. However, after 16 hours at 2600°F, a FCC phase, most likely Hf(CN), is beginning to form. Accompanying the formation of the Ta_2C and Hf(CN) precipitate is a drop in the room temperature hardness (See Figure 11). The largest change in hardness occurred after the first hour which is indicative of the rapid precipitation kinetics of the carbide precipitation reaction. The room temperature hardness after aging for 16 hours at 2000, 2200, and 2400°F is similar. However there is a definite increase in the hardness level for specimens aged at 2600°F which may reflect the higher interstitial solubility or may be related to the nitride precipitation reaction. The appearance of the FCC phase was not unexpected since a similar composition tested early in this investigation exhibited the same behavior. (9)

III. FUTURE WORK

During the next period the following will be accomplished.

1. Initiate creep testing of controlled grain size creep specimens.

2. Continue investigation on the effect of long time annealing treatments on the phase morphology and stability of the ASTAR-811C and ASTAR-811CN.

3. Complete 500 hour annealing treatment at 2200°F on TIG welded ASTAR-811CN (Heat NASV-23) and determine ductile brittle transition temperature.

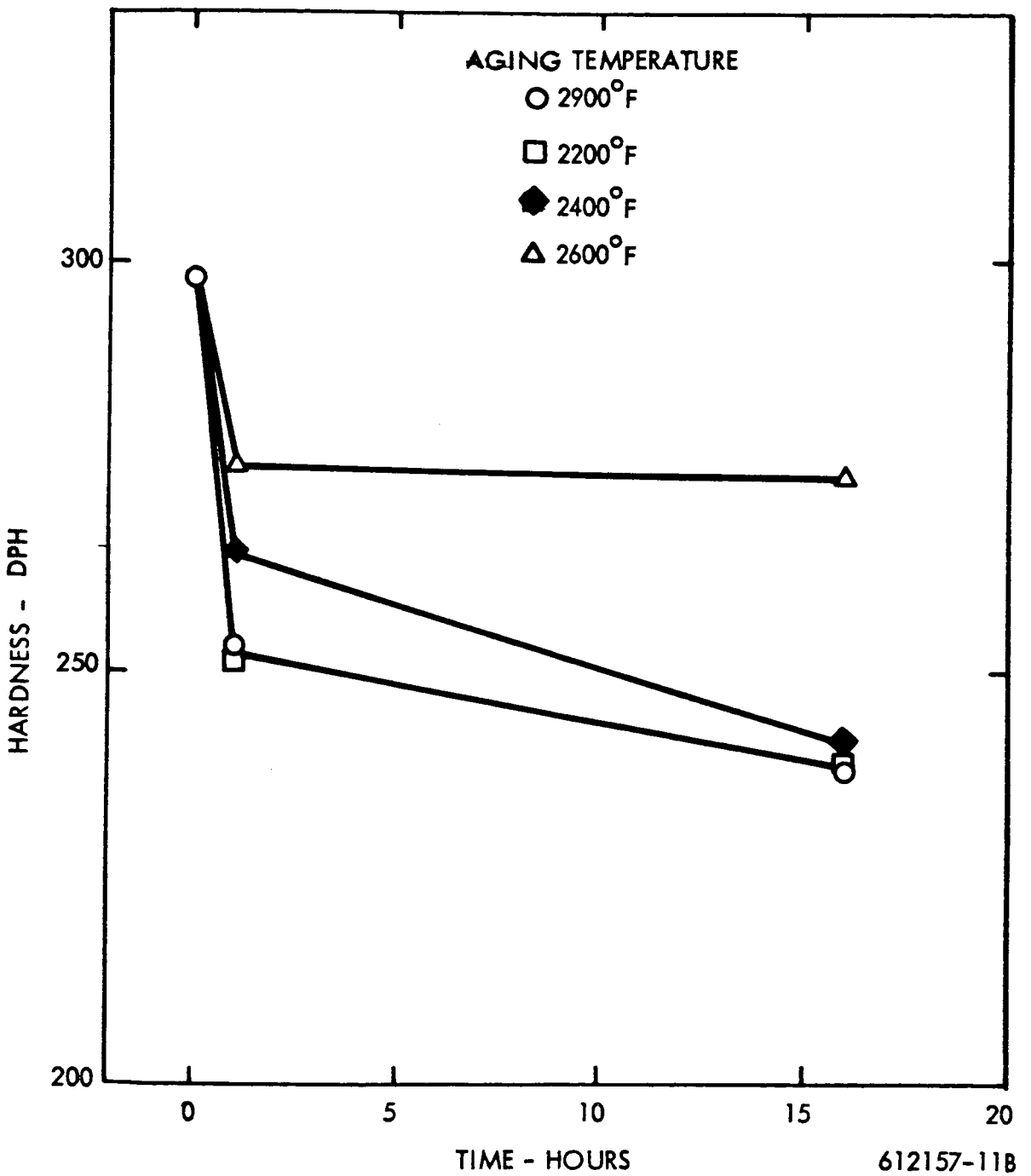


FIGURE 11 - Aging Behavior of ASTAR-811CN
(Annealed 1 Hr. at 3000°F Prior to Aging)

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