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VANADIUM

PURIFICATION

EIGHTH QUARTERLY PROGRESS REPORT

Issued November, 1968

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UC-25, Metals, Ceramics
and Materials
TID-4500, 51st Edition

VANADIUM PURIFICATION EIGHTH QUARTERLY PROGRESS REPORT

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VANADIUM PURIFICATION
Eighth Quarterly Progress Report

Table of Contents

<u>Section</u>		<u>Page No.</u>
	Abstract	ix
I	Introduction	1
II	Experimental Effort	2
	A. Tensile Test Results	3
	B. Stress-Rupture Test Results	3
III	Discussion	9
IV	Future Work	16

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List of Tables

<u>Table No.</u>		<u>Page No.</u>
I	Material Production and Distribution	3
II	Tensile Results on V-15Cr-5Ti Alloy	4
III	V-15Cr-5Ti Stress-Rupture Data	6

VANADIUM PURIFICATION
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List of Illustrations.

<u>Figure No.</u>		<u>Page No.</u>
1.	Mechanical Test Results 1050°C/1 hr. Annealed (Recrystallized) V-15Cr-5Ti	5
2.	650°C Stress-Rupture Tests, V-15Cr-5Ti Alloy Sheet Recrystallized	7
3.	800°C Stress-Rupture Tests, V-15Cr-5Ti Alloy Sheet Recrystallized	8
4.	Time to Rupture Results, V-15Cr-5Ti Alloy Sheet	10
5.	Minimum Creep Rate, V-15Cr-5Ti Alloy Sheet	11
6.	Time to 3rd Stage Creep	12
7.	Stress Rupture and Tensile Specimen	13
8.	Comparison of ANL and CENPD Creep Data	14

ABSTRACT

During this report period the second batch of V-15Cr-5Ti sheet was fabricated, making a total of 45.6 pounds of sheet produced.

The alloy ingot for use at Argonne National Laboratory was shipped, completing the material production phase of the program.

Tensile testing of the recrystallized sheet is complete along with eight stress rupture tests. The material gives typical curves for time-to-rupture and minimum-creep-rate properties.

I. INTRODUCTION

This program was initiated to produce a 100-pound, high purity (<500 ppm total interstitial content) vanadium ingot by a process suitable for commercial applications. The process developed by Wah Chang, Albany, Oregon, to produce the ingot is as follows:

- 1) Aluminothermic reduction of V_2O_5 ;
- 2) Consolidation by electron beam melting the vanadium reductions;
- 3) Double electron beam melting.

The high purity vanadium ingot was then alloyed by:

- 1) Compaction of vanadium, titanium and chromium flakes and chips;
- 2) Double arc melted, first in argon, then in vacuum.

Combustion Engineering is presently evaluating the high purity (~500 ppm interstitial) V-15Cr-5Ti alloy sheet. The purpose of this evaluation is to determine the effect of lower interstitial content on the mechanical properties of this alloy. Argonne National Laboratory is in the process of determining the mechanical properties on the same alloy with an interstitial content of approximately 1200 ppm. The present program will provide comparison of short term tensile and stress-rupture data on both base metal and welded sheet material.

Argonne National Laboratory, in their evaluation of the lower purity material, found that recrystallization occurred after annealing at 900°C for 1 hour with a resulting grain size of ASTM-9 to 10. Heat treating at higher temperatures produced little if any change in grain size up to 1400°C. Above this temperature very rapid grain growth occurred. It is assumed that the abrupt increase in grain size was due to solutioning of grain boundary precipitates

above 1400°C. Argonne National Laboratory chose to anneal their material at 1250°C for 1 hour for optimum stress rupture properties.

Heat treat studies on the higher purity material showed complete recrystallization after 1 hour at 1050°C. The grain size after this treatment was ASTM-7 to 8. Annealing above 1050°C caused initiation of grain growth. This differed from the Argonne National Laboratory results, and is assumed to be related to insufficient grain boundary impurities in the higher purity material to retard grain growth. Heat treating at 1250°C for 1 hour resulted in a grain size of ASTM-4 to 5, as compared to approximately ASTM-8 for the Argonne National Laboratory material.

Based on the above heat treating studies, it was decided to test the high purity material in the 1050°C and 1250°C annealed conditions. The majority of the tests will be performed on recrystallized (1050°C) specimens since this would more nearly duplicate the structure and grain size of the Argonne National Laboratory tests. A limited number of tests will be performed at 1250°C to evaluate the effects of grain size and second phase on strength properties.

II EXPERIMENTAL EFFORT

During this quarter, the second batch of V-15Cr-5Ti sheet was rolled at Wah Chang, Albany, Oregon, making a total of 45.6 pounds of sheet. The process for this batch is similar to that described in the preceding quarterly report, CEND-3742-342. The weight of total material produced for the program is summarized below in table 1.

Table I. Material Production and Distribution

Material	Form	Weight, lb	Date Shipped	Recipient
V-15Cr-5Ti	0.040" sheet	20.0	6-4-68	C-E
V-15Cr-5Ti	0.040" sheet	13.6	7-24-68	C-E
V-15Cr-5Ti	3.7" billet	23.0	8-13-68	ANL
Vanadium	Scrap	92.0	8-21-68	Bureau of Mines
Vanadium	Scrap	9.2	8-26-68	Bureau of Mines
V-15Cr-5Ti	Scrap	41.3	8-26-68	Bureau of Mines
		199.1 lbs		

A. TENSILE TEST RESULTS

Results from tensile tests performed on the recrystallized (1050°C/1 hour) material are shown in table II and figure 1. All tests were performed in a purified argon atmosphere at a strain rate of 0.05 in./in./min. The increase in strength and decrease in elongation at approximately 625°C is probably due to a strain aging mechanism. All elevated temperature tensile tests showed upper and lower yield points similar to that observed by Argonne National Laboratory with tests on V-10Ti and V-20Ti material. Typical stress-strain curves were shown in the preceding quarterly report.

No tensile tests have been performed to date on the 1250°C annealed material.

B. STRESS-RUPTURE TEST RESULTS

Stress-rupture tests at 650°C and 800°C have been initiated on recrystallized (1050°C/1 hour) specimens. Table III summarizes the data generated to date. Figures 2 and 3 show strain versus time curves for the eight stress-rupture tests completed. Stress-rupture tests of 10, 100 and 500 hours are the target

Table II. Tensile Results on V-15Cr-5Ti Alloy*

(0.040" thick sheet)

Test Temperature and Condition	Ultimate Tensile Strength (psi)	0.2% Yield Strength (psi)	Upper Yield Strength (psi)	Lower Yield Strength (psi)	Reduction of Area (%)	Elongation (%)
As-Received	131,700	129,300	--	--	18.9	4.5
Room Temperature	140,000	133,100	--	--	18.0	5.5
(70% cold worked)	<u>132,000</u>	<u>128,900</u>	--	--		
Average:	134,600	130,400			18.5	5.0
As-Received	111,400	101,600	--	--	6.7	5.0
600°C Test Temp.	<u>115,400</u>	<u>95,600</u>	--	--	<u>7.2</u>	<u>4.0</u>
Average:	113,400	98,600			6.9	4.5
1050°C/1 hour	86,900	--	73,600	73,200	45.6	25.0
Room Temperature	88,400	--	76,900	75,900	46.5	25.0
	<u>87,100</u>	--	<u>77,600</u>	<u>73,500</u>	<u>47.2</u>	<u>28.0</u>
Average:	87,500		76,000	74,200	46.4	26.0
1050°C/1 hour	74,400	--	48,000	46,500	34.6	17.0
500°C Test Temp.	<u>71,700</u>	--	<u>47,100</u>	<u>44,900</u>	<u>34.7</u>	<u>16.0</u>
Average:	73,100		47,600	45,700	34.7	17.0
1050°C/1 hour	74,200	--	46,400	45,200	39.7	15.0
600°C Test Temp.	75,600	--	47,400	44,500	32.8	14.0
	<u>76,300</u>	--	<u>47,700</u>	<u>45,100</u>	<u>36.9</u>	<u>14.0</u>
Average:	75,300		47,200	44,900	36.5	14.0
1050°C/1 hour	74,800	--	50,400	47,400	43.8	20.0
700°C Test Temp.	<u>74,700</u>	--	<u>46,000</u>	<u>45,300</u>	<u>38.0</u>	<u>18.5</u>
Average:	74,800		48,200	46,900	40.9	19.2
1050°C/1 hour	58,800	--	38,500	38,100	43.9	25.0
800°C Test Temp.	59,200	--	40,000	39,600	43.8	23.0
	<u>59,500</u>	--	<u>40,000</u>	<u>40,000</u>	<u>50.1</u>	<u>24.0</u>
Average:	59,200		39,500	39,200	45.9	24.0

*Interstitial Content (ppm) $\frac{C}{100}$ $\frac{N}{90}$ $\frac{O}{100}$ $\frac{H}{4}$

Figure 1.
MECHANICAL TEST RESULTS 1050°C/ 1 hr ANNEALED (RECRYSTALLIZED)
V-15Cr-5Ti

Table III. V-15Cr-5Ti Stress-Rupture Data

Temp °C	Stress ksi	Time to Rupture Hrs	Time to 3rd Stage Hrs	Min Creep Rate in./in./hr	Initial ε %	ε Intercept %	ε at 3rd Stage %	Final ε %	Hardness Traverse to Center- line* DPH	Test No.
650	74.0	3.0	1.3	4.6×10^{-2}	11.3	11.4	4.6	25.0	274 272 266	CR 5
650	72.5	5.6	2.7	2.2×10^{-2}	11.2	11.5	14.0	22.0	249 245 242	CR 6
650	65.0	58.8	33.0	1.0×10^{-3}	5.1	5.6	7.1	18.5	255 242 238	CR 7
800	50.0	1.4	0.5	6.1×10^{-2}	1.2	2.4	4.6	34.0	251 255 262	CR 3
800	40.0	28.1	12.0	4.0×10^{-3}	0.2	0.8	3.2	21.0	548 240 235	CR 4
800	37.5	28.2	8.5	1.4×10^{-2}	0.1	0.4	2.0	18.0	220 219 216	CR 8
800	35.0	118.3	28.0	5.8×10^{-4}	0.03	0.6	2.0	16.5	425 219 219	CR 9
800	35.0	72.0	27.0	1.0×10^{-3}	0.03	0.6	4.0	23.5	294 234 219	CR 10

* As Annealed Sheet

211
204
201

values set for the program.

Time-to-rupture and minimum creep rate data as a function of stress are presented in figures 4 and 5, respectively. It can be seen that the data points show good agreement.

Time to third stage creep versus stress is shown in figure 6 and indicates no anomalies.

All tests have been run in static purified argon atmosphere. The argon is purified by passing it through a titanium sponge at 1600°F. In addition, tantalum foil is loosely wrapped around the sample to act as a getter. Strain is measured by a means of a dial gage attached to the lever arm of the creep machine.

The load is applied to the stress-rupture sample in 5 ksi increments, enabling an initial loading curve (stress-strain) to be plotted.

When computing strain values from dial gage readings, the 2-inch gage length of the sample (see figure 7) is considered to have a 2.3-inch effective gage length. This is necessary since the dial gage measures the total elongation of the sample, not just the 2-inch gage length. This has been demonstrated to be quite accurate when the dial gage readings near the fracture point are compared to measurements taken from the fractured samples.

III DISCUSSION

Only one stress-rupture test on the lower purity Argonne National Laboratory (ANL) material is presently available for comparison. A test was conducted by ANL at 700°C at 42,000 psi and was not run to failure but was terminated after 300 hours. The minimum creep rate was approximately 1.6×10^{-7} in./in./hour, which falls between the 650°C and 800°C results shown in figure 8. There are insufficient data to comment on the relative strengths or creep properties of the two materials at this time.

Figure 4
TIME TO RUPTURE RESULTS
V-15Cr-5Ti ALLOY SHEET
ANNEALED ONE HOUR AT 1050°C
ALL TESTS PERFORMED IN ARGON ATMOSPHERE

Figure 5

MINIMUM CREEP RATE
V-15 Cr -5Ti ALLOY SHEET
ANNEALED ONE HOUR AT 1050°C
ALL TESTS PERFORMED IN ARGON ATMOSPHERE

Figure 6
TIME TO 3RD STAGE CREEP

Figure 7
STRESS RUPTURE AND TENSILE SPECIMEN

Figure 8
COMPARISON OF ANL AND CENPD CREEP DATA

Stress-rupture tests conducted at 650°C required loadings in excess of the 0.2 percent yield strength to cause failure within 100 hours. Table II shows that considerable strain occurred on initial loading of these specimens, 5 to 11 percent. The material at this temperature is yield strength dependent. The tests to date indicate that the 650°C, 100 hour rupture strength is approximately 63,000 psi. In all cases, failure has been transgranular with failure ductility in the range of 19 to 25 percent.

Post test hardness values on the 650°C and 800°C stress-rupture samples showed a possible aging effect. Centerline hardness of the 650°C samples showed values of 266, 242 and 238 DPH. These values represent times at 650°C of 3.0, 5.6 and 58.5, respectively. Vacuum aging studies are presently being performed to verify this effect.

The 800°C stress-rupture tests produced a 100-hour rupture strength of approximately 35,000 psi, 87.5 percent of the yield strength. Again, failure was transgranular. Two of the test specimens (CR-4 and CR-9) showed evidence of contamination on the surface after test. Micro hardness measurements show a case less than 5 mils in depth on both samples. The presence of the surface contamination did not appear to affect the creep or rupture data shown in figures 4 and 5. Chemical analyses for oxygen, nitrogen, carbon and hydrogen are being performed on all the stress-rupture samples.

The fact that the contaminated specimens showed no difference in creep characteristics is surprising. It would be expected that the minimum creep rate and rupture time would be affected. It has been suggested by ANL that this alloy may exhibit an ordered structure. The extra activation energy, needed to move super lattice dislocations necessary to preserve order across the slip plane,

might mask the effect of interstitial contamination. These dislocations will not be able to cross-slip or climb readily and can greatly influence the creep rate. This is borne out by the high resistance to creep at test temperatures of $0.45 T_m$ (650°C) and $0.53 T_m$ (850°C). At these temperatures, the role of solute atom locking in steady state creep can be expected to be small.

IV FUTURE WORK

Tensile tests will be performed on the 1250°C annealed material and stress-rupture tests on the 1050°C annealed material will be extended to 500 hours. Electron beam and TIG welded samples are presently being prepared using the low interstitial material. Short time tensile, stress-rupture and aging studies will be conducted on these samples.

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